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치의학박사학위논문

Effect of Various Surface Modifications on the Optical Properties of Dental Monolithic Zirconia Restorations

다양한 표면 변화가 치과용 단일 구조 지르코니아 수복물의 광학적 특성에 미치는 효과에 관한 연구

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치의과학과 치과보철학전공

김 희 경

Effect of Various Surface Modifications on the Optical Properties of Dental Monolithic Zirconia Restorations

2014

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*(Directed by Prof., **Jai-Bong Lee,** DDS, MSD, PhD)*

CONTENTS

ABSTRACT

Purpose. The purpose of this study was to investigate the effect of various surface modifications on the optical properties of monolithic zirconia restorations. Experiment I investigated the effect of the number of coloring liquid applications on the optical properties of monolithic zirconia. Experiment II investigated the effect of polishing and glazing on the color and translucency of monolithic zirconia. Experiment III investigated the effect of the amount of thickness reduction on the color and translucency of monolithic zirconia.

Material and Methods. BruxZir monolithic zirconia blocks and Tanaka ZirColor A2-coloring liquid were used for this study. Eighteen monolithic zirconia specimens (27.6 mm × 27.6 mm × 2.0 mm) were prepared for Experiment I and divided into 6 groups (n = 3) according to the number of coloring liquid applications (Group I to V) and Group O as a control. Color coordinates (CIE L^*, a^* and b^* value), color difference (ΔE^*_{ab}), translucency parameter (TP) and opalescence parameter (OP) were recorded. Forty-five monolithic zirconia specimens (16.3 mm × 16.4 mm × 2.0 mm) were prepared for Experiment II and divided into 5 groups according to the number of coloring liquid applications (Group I to V). Each group was divided into 3 subgroups according to the method of surface treatments (n = 3): N: no

treatment; P: polishing; G: glazing. CIE L^*, a^* and b^* value, ΔE^*_{ab} and TP were recorded. One-hundred sixty-five monolithic zirconia specimens (16.3 mm × 16.3 mm × 2.0 mm) were prepared for Experiment III and divided into 5 groups according to the number of coloring liquid applications (Group I to V). Each group was divided into 11 subgroups according to the amount of thickness reduction in 0.1-mm increments until final thickness was 1.0 mm (n = 3): Subgroup 0: no reduction; Subgroup 1: 0.1 mm reduction; Subgroup 2: 0.2 mm reduction; Subgroup 3: 0.3 mm reduction; Subgroup 4: 0.4 mm reduction; Subgroup 5: 0.5 mm reduction; Subgroup 6: 0.6 mm reduction; Subgroup 7: 0.7 mm reduction; Subgroup 8: 0.8 mm reduction; Subgroup 9: 0.9 mm reduction; Subgroup 10: 1.0 mm reduction. CIE L^*, a^* and b^* value, ΔE^*_{ab}, TP value were recorded. All measurements were performed on five different areas of each specimen according to CIELAB color space relative to the standard illuminant D65 on a reflection spectrophotometer. Data were analyzed using one-way ANOVA followed by multiple comparison Scheffé test, Pearson correlation and regression analysis (α = 0.05).

Results. According to the results of Experiment I, with the increase of the number of coloring liquid applications, CIE L^* and OP values decreased, while CIE b^* increased. Color differences among groups ranged from 1.3 to 15.7 ΔE^*_{ab} units. TP values were not significantly changed. Experiment II showed that there was a significant difference in CIE L^* between Subgroup

N and P, and in CIE b^* between Subgroup P and G in all groups. A perceptible color difference was obtained between Subgroup N and P in all groups and between Subgroup N and G in most groups ($\Delta E^*_{ab} > 3.7$). There was no significant difference in TP values between each subgroup in most groups. Experiment III showed that thickness reduction resulted in the decrease in CIE L^* and b^* values and the increase in a^* values. Perceptible color difference was obtained between Subgroup 0 and Subgroup 1 ($\Delta E^*_{ab} > 3.7$) in all groups. TP values generally increased with the increase of thickness reduction in all groups.

Conclusion. Within the limitations of this study, the following conclusions can be drawn. Increasing the number of coloring liquid applications results in a decrease of the lightness and an increase of the yellowish appearance of monolithic zirconia restorations. Polishing decreases the lightness and glazing also decreases the lightness, while it increases the yellowish appearance of monolithic zirconia restorations. Increasing thickness reduction decreases lightness and increases the reddish appearance and decrease yellowish appearance of monolithic zirconia restorations. Reduced thickness of monolithic zirconia produces more translucent monolithic zirconia restorations.

Keyword: Y-TZP ceramic; Color; Translucency; Dental Prosthesis Coloring; Surface modifications
Student number: 2003 – 31121

DECLARATION

I declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.

DEDICATION

To my family, Yong-Ho, Jin-Yung, Sung-Joon

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1. INTRODUCTION

Metal-ceramic restorations have been introduced since 1962 [1] and widely used in fixed dental prostheses for many decades as an esthetic restoration. For metal-ceramic restorations, light reflection from an opaque porcelain layer to mask the metal substrate resulted in opaque appearance and thereby, their use in high esthetic area was limited [2]. All-ceramic restorations without metal substrate induced more light transmission within the crown and accordingly, they improved the ability to reproduce the appearance of natural tooth [3]. However, brittleness was exhibited as a major drawback with being limited in a clinical use of all-ceramics [4]. Since the advent of high strength zirconia [5], zirconia-based material combined with CAD/CAM technology has broaden the range of their applications in dentistry. Due to its whitish and opaque color, it has to be veneered with feldspathic porcelain for more acceptable esthetic outcome, but cohesive failure of the veneering porcelain has been identified as a main complication [6, 7]. Several factors that reduce the cohesive failure of veneering porcelain have been studied. Fabricating optimized zirconia substructures [8], matching of thermal expansion between core and veneering ceramics [9], uniform thickness of cement layer [10], reduced thickness ratio of veneering porcelain [11], increased cooling rate [12] could reduce the incidence of cohesive veneering failure. Furthermore, in an attempt to reinforce the veneering porcelain, several trials such as high

strength CAD/CAM-fabrication of veneering porcelain [13], high strength heat-pressed ceramic [14], and "double veneering" technique [15] have been performed. Other approach to control the veneering failure could be a fabrication of monolithic zirconia which consists of a single zirconia material without any veneering. Nowadays the advantages of monolithic zirconia restorations with an increased mechanical stability make them possible to expand their clinical indications [16]. In addition to mechanical advantage of monolithic zirconia, this new approach also requires esthetic considerations.

Color and translucency can be determined from the transmission or reflection of the light that is absorbed, scattered, refracted and reflected [17]. Polycrystalline contents of zirconia induce maximum light scattering and diffuse reflection and thereby, result in opaque appearance to visible light [18]. Due to its inherent whitish and opaque appearance, the overall appearance of monolithic zirconia can be improved by surface modifications, such as coloring procedure, surface polishing and glazing. Unlike metal-ceramic restorations, monolithic zirconia can be colored in a pre-sintered state to match adjacent teeth.

There are common surface treatment methods for ceramic restorations, such as polishing and glazing. Sequential polishing procedures using various diamond points, rubber wheels, and abrasive pastes may give a luster to the

surface [19]. Glazing can be created either by firing a small coating of transparent glass onto the surface or by heating the restoration up to glazing temperature for 1 or 2 minutes to get shiny gloss surfaces [19]. Several studies [20-22] have compared glazing with different polishing techniques for ceramic restorations regarding surface texture. They demonstrated that polishing on feldspathic porcelain could be used as an alternative method for glazing. Other studies [23-25] investigated the effect of surface treatments on the color of porcelain restorations. According to these studies, surface treatments including polishing and glazing could affect the color of porcelain restorations. However, there have been no reported studies that investigated the effect of surface polishing and glazing on the color and translucency of monolithic zirconia.

The surface might also be modified in the adjustment procedures by the dentist to achieve an optimal occlusal contact. Grinding the ceramic surface with a diamond bur results in the thickness reduction of monolithic zirconia restorations. There have been several studies to investigate the importance of the ceramic thickness on overall color and translucency of ceramic restorations. However, those studies investigated the change of overall color of ceramic restorations with regard to masking effect of underlying substrate [26], core/veneer interaction [27, 28] and dentin porcelain overlying opaque substrate [2, 25, 29] based on metal-ceramic or all-ceramic restorations and

did not address the color change of material itself as a function of changes in thickness. Another previous study [30] determined the change of contrast ratio as a parameter for translucency measurements with the different thickness of all-ceramic core and veneering materials, respectively, to predict the translucency of multi-layered ceramic structures as a function of changes in thickness. However, there have been no reported studies regarding the change of color and translucency as a function of changes in thickness using monolithic zirconia which is a single-layered ceramic structure.

Any surface modifications of monolithic zirconia restorations might induce the shift of esthetic outcomes in terms of optical properties. In addition, there would be a significant implication of opalescence property concerning clinical application of monolithic zirconia due to its white and opaque appearance. The purpose of Experiment I was to investigate the effect of the number of coloring liquid applications on the color, translucency and opalescence of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in the optical properties between monolithic zirconia ceramics with the different number of coloring liquid applications. The purpose of Experiment II was to investigate the effect of polishing and glazing on the color and translucency of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in color and translucency parameters between monolithic zirconia ceramics

with the different surface treatments. The purpose of Experiment III was to evaluate the effect of the amount of thickness reduction on the color and translucency of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in color and translucency parameters between monolithic zirconia ceramics with different amounts of thickness reduction.

2. MATERIAL and METHODS

Monolithic zirconia (BruxZir, yttria-stabilized tetragonal zirconia polycrystal, Glidewell Laboratories, Newport Beach, CA, USA) was investigated in this study (Table 1). Tanaka ZirColor of A2 shade (Tanaka Dental, Skokie, IL, USA) was used as a coloring liquid (Table 1). It is designed to be brushed on and dried quickly with no drying time between each application and, thus no preheating is necessary before sintering.

2.1. Specimen preparation and optical properties measurement

2.1.1. ***Experiment I****. Effect of the number of coloring liquid applications on the optical properties of monolithic zirconia*

Eighteen square-shaped, pre-sintered zirconia block (34.0 mm × 34.0 mm × 2.7 mm) were fabricated using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a synthetic nylon fiber brush (No.156, Hwahong, Hwasung-si, Kyunggi-Do, Korea; Fig. 1). These specimens were divided into six groups (n = 3) according to the number of coloring liquid applications. The specimen with no application was used as a control.

- Group O (Control group): No application
- Group I: One time of application

Table 1. Materials investigated

Type	Brand name	Composition	Lot No.		Manufacturer
Monolithic zirconia block	BruxZir	Yttria-stabilized tetragonal zirconia polycrystal	Exp. I Exp. II Exp. III	B 186338 B 84942 B 105566 B 105583 B 105565 B 88712	Glidewell Laboratories, Newport Beach, CA, USA
Coloring liquid	Tanaka ZirColor (A2 shade)	(R)-p-mentha-1,8-diene, 50-75% Stoddard solvent, 10-25%	Exp. I Exp. II Exp. III	50297 50003 40127 40129	Tanaka Dental, Skokie, IL, USA

• Exp: Experiment

Fig. 1. (A) Tanaka ZirColor coloring liquid of A2 shade was used. (B) The coloring liquid was brushed on with a brush according to the manufacturer's recommendations.

- Group II: Two times of application
- Group III: Three times of application
- Group IV: Four times of application
- Group V: Five times of application

All specimens were then fired in a zirconia sintering furnace (LHT 0217, Nabertherm GmbH, Bahnhofstr, Germany). The sintering cycle was controlled as follows: The temperature was raised to 950°C for 1.5 hours and maintained for 2 hours, and then raised up to 1550°C for 1.5 hours and maintained for 3 hours. After sintering process, the shrinkage of specimens was *circa* 20%. The mean dimension of sintered specimens was 27.6 mm × 27.6 mm (Fig. 2), verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The thicknesses of specimens were 1.934 ± 0.059 mm. Colored surface of the specimen was neither grinding nor polishing after completion of sintering. All

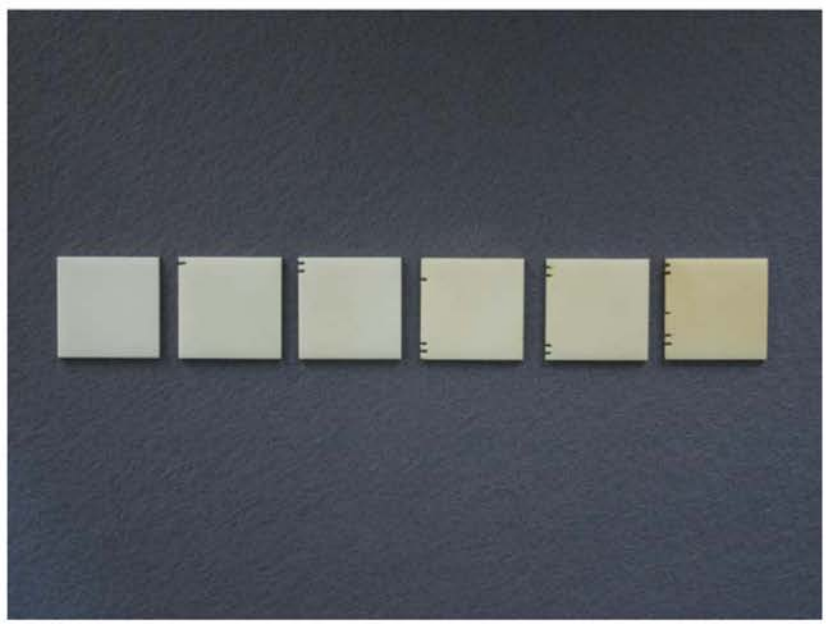

Fig. 2. Colored specimens of each group after sintering process. The number of slots on the left side indicated the number of coloring liquid applications (Experiment I).

specimens were ultrasonically cleaned in distilled water for 5 minutes before testing.

Color and spectral distribution were taken with a double-beam spectrophotometer (Cary 5000 UV-VIS-NIR Spectrophotometer, Agilent Technologies Inc., Santa Clara, CA, USA) (Fig. 3) using an integrating sphere attachment. The specular reflectance component was excluded (SCE mode) by gloss trap inserted. Relative reflectance data was recorded in the visible range from 380 to 780 nm at 5 nm intervals. Measurements were recorded in Commission Internationale de l'Eclairage (CIE) 1976 $L^*a^*b^*$ color space (CIELAB, Fig. 4) relative to the standard illuminant D65 in the reflectance mode over a white background (CIE $L^* = 99.9701$, $a^* = -0.0711$ and $b^* = 0.0499$) and a black background (CIE $L^* = 4.7487$, $a^* = -1.6749$ and $b^* = -1.5844$) and in the transmittance mode. The white standard was polytetrafluoroethylene (PTFE) plate (SRS-99-020, Spectralon® Reflectance Standards, Labsphere Inc., North Sutton, NH, USA) and the black background was a black tile (CM-A101B, Konica Minolta Optics Inc., Tokyo, Japan). The spectrophotometer was equipped with an integrating sphere of 150 mm diameter made with sintered PTFE. The geometry for the reflectance measurements was *8°:de* (eight degree: diffuse geometry, specular component excluded). Thus, CIE 1964 10° supplementary standard observer,

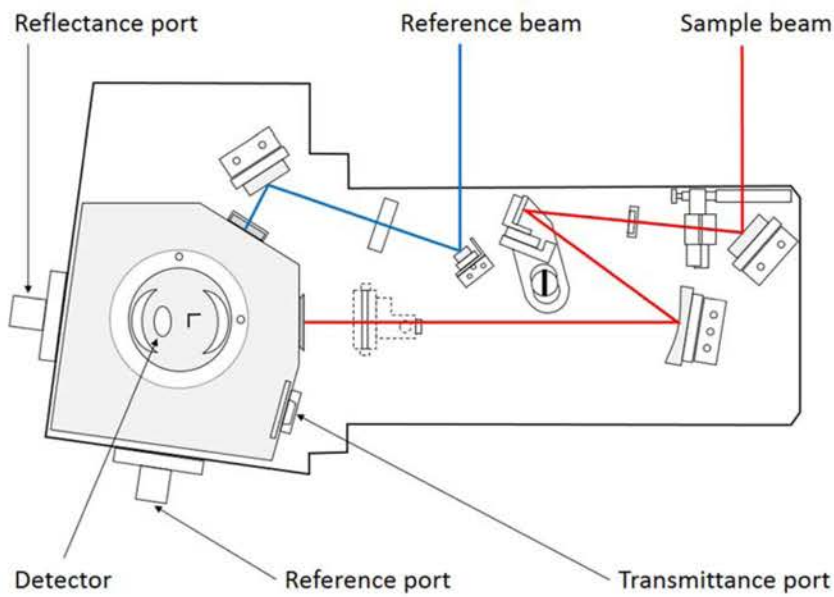

Fig. 3. A schematic view of double-beam spectrophotometer (Cary 5000 UV-VIS-NIR Spectrophotometer, Agilent Technologies Inc., Santa Clara, CA, USA).

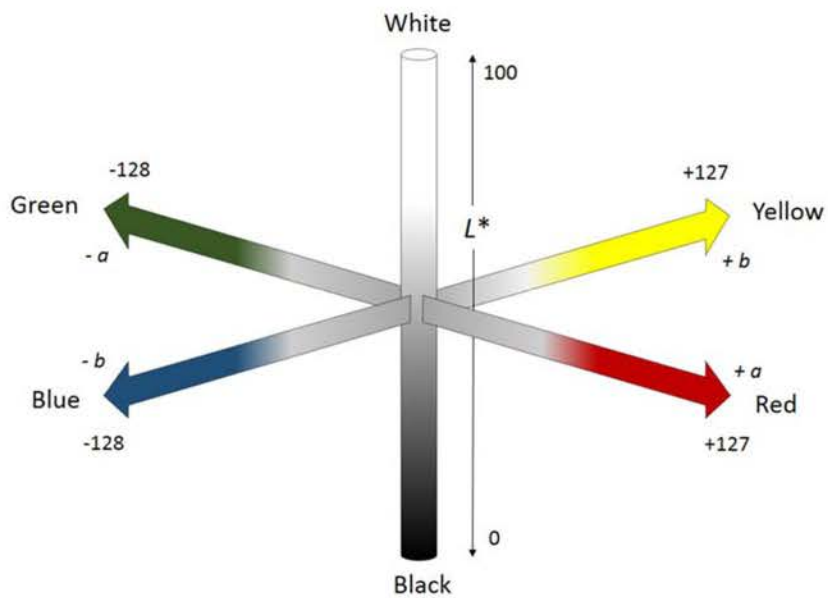

Fig. 4. CIE 1976 $L^*a^*b^*$ color space. The three coordinates of CIELAB represent the lightness of the color (L^*), its position between red and green (a^*) and its position between yellow and blue (b^*).

which is more modern and alternative to CIE 1931 2° standard observer, was selected. The aperture size was 19 mm in diameter for the reflectance measurement. For the transmittance measurement, opaque black polyvinyl chloride (PVC) plate supported measuring aperture to make the aperture size 10 mm × 15 mm, because the original aperture size of the instrument was 10 mm × 35 mm for the transmittance measurement. The specimens of 27.6 mm × 27.6 mm for this study provided adequate area for color measurement. The white PTFE standard was used for zero/base correction before reflectance color measurement.

Color coordinates, CIE L^*, a^* and b^*, were determined from the transmittance and reflectance data using a computer software (Cary WinUV Software, Agilent Technologies Inc., Santa Clara, CA, USA). Since the beam size was 1 mm × 5 mm, an effort was made not to overlap the beam on each measurement. Average L^*, a^* and b^* values were used to calculate CIE 1976 a,b (CIELAB) color difference, ΔE^*_{ab} of each group set using the following equation (Eq. 1) [31].

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad \text{(Eq. 1)}$$

where, the L^* coordinate represents the lightness of an object, the a^* value represents the red or green chroma, and the b^*value represents the yellow or

blue chroma and ΔL^*, Δa^* and Δb^* indicate the differences between the CIE L^*, a^* and b^* color parameters of two specimen groups.

The translucency parameter (TP) was obtained by calculating the color difference of the specimen over the black and white backgrounds with the following equation (Eq. 2) [32].

$$TP = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2} \quad \text{(Eq. 2)}$$

where, subscript B refers to the color coordinates over a black background and the subscript W refers to those over a white background. TP value of zero corresponds to a completely opaque material. The greater the TP value, the higher the actual translucency of the material.

The opalescence parameter (OP) was calculated as the difference in yellow-blue and red-green coordinates between the transmitted and reflected colors using the following equation (Eq. 3) [33].

$$OP = [(\text{CIE } a_T^* - \text{CIE } a_R^*)^2 + (\text{CIE } b_T^* - \text{CIE } b_R^*)^2]^{1/2} \quad \text{(Eq. 3)}$$

where, subscript T refers to the transmitted color and subscript R refers to the reflected color over a black background. All measurements were performed

on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each group were obtained.

2.1.2. ***Experiment II****. Effect of polishing and glazing on the color and translucency of monolithic zirconia*

Forty-five square-shaped, pre-sintered zirconia blocks (20.0 mm × 20.0 mm × 2.7 mm) were prepared using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a brush (2850-B, Babara, Kobe, Japan). These specimens were divided into five groups according to the number of coloring liquid applications.

- Group I: One time of application
- Group II: Two times of application
- Group III: Three times of application
- Group IV: Four times of application
- Group V: Five times of application

All specimens were then sintered in a zirconia sintering furnace (LHT 0217, Nabertherm GmbH, Bahnhofstr, Germany). The sintering cycle was controlled following the protocol of Experiment I. After sintering process,

shrinkage of specimens was *circa* 20%. The mean dimension of sintered specimens was 16.3 mm × 16.4 mm (Fig. 5), verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The specimen thicknesses were 1.990 ± 0.025 mm. Nine specimens of each group were assigned to three subgroups according to the surface treatment (n = 3).

- Subgroup *-N (Control group): No treatment
- Subgroup *-P: Polishing
- Subgroup *-G: Glazing

*: I, II, III, IV or V

In Subgroup P, specimens were polished with a sequence of three diamond-impregnated silicone discs for 60 seconds each. Then, specimens were polished using a felt wheel with diamond polishing paste for 60 seconds each

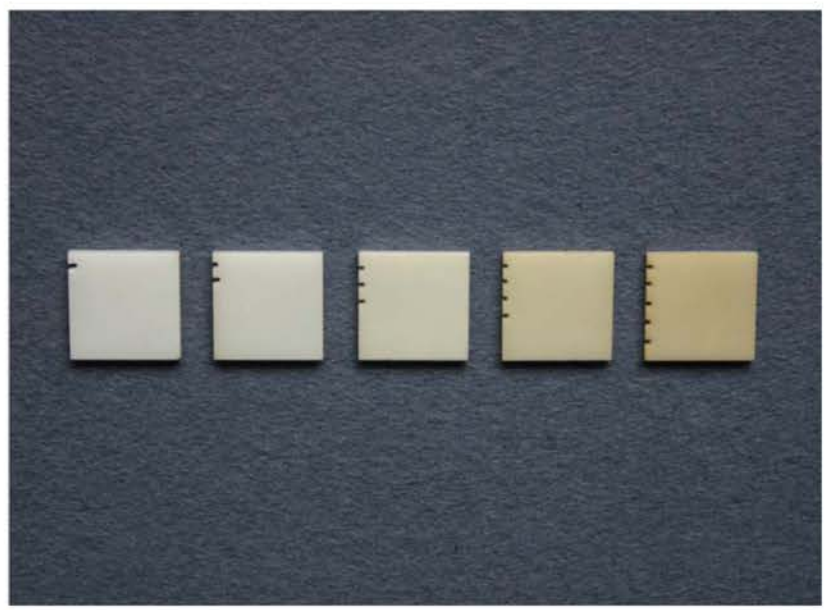

Fig. 5. Colored specimens of each group after sintering process. The number of slots on the left side indicated the number of coloring liquid applications (Experiment II).

consecutively (Fig. 6). Details of polishing instruments were described in Table 2.

In Subgroup G, specimens were glazed in vacuum in a ceramic furnace (Austromat 3001, DEKEMA Dental-Keramiköfen GmbH, Freilassing, Germany) using a glazing paste (Fig. 7, Table 3) for each, following the protocol: The temperature was raised up to 950°C at the firing rate of 30°C/min, and maintained for 30 seconds, and then cooling down to 300°C at 15°C/min.

Subgroup N indicating no surface treatment served as a control. All specimens were ultrasonically cleaned in distilled water for 5 minutes before testing.

Colors were measured according to CIELAB color space in the reflectance mode relative to the standard illuminant D65 on a reflection spectrophotometer (Fig. 8), which was equipped with an integrating sphere. Illuminating and viewing configurations of this instrument were *de:8°* geometry and the 10° CIE 1964 supplementary standard colorimetric observer was selected. The aperture diameter of the measuring port of the spectrophotometer (Target Mask CM-A 121, Minolta, Osaka, Japan) was 3 mm. White calibrating plate (CM-A120, Minolta, Osaka, Japan) was

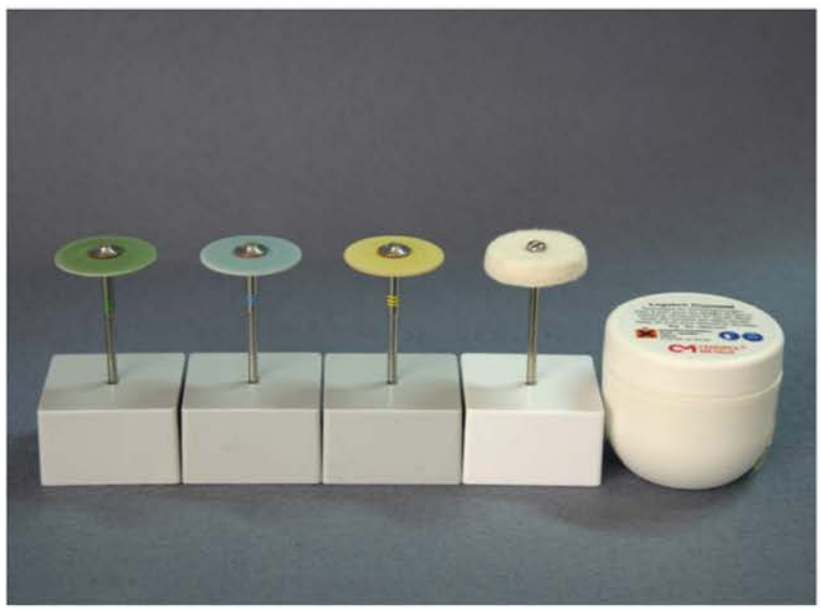

Fig. 6. Three diamond impregnated silicone discs (green: coarse grit, blue: medium-coarse grit, yellow: super-fine grit, Edenta AG, Au SG, Switzerland) and felt wheel (Super-Snap Buff, Shofu Inc., Kyoto, Japan) with diamond polishing paste (LegabrilDiamond, Metalor Dental AG, Biel/Bienne, Switzerland) (Experiment II).

Table 2. Polishing instruments used

Instruments	Lot No.	Grit/Contents	RPM	Manufacturer
CeraGloss 310 HP (Green)	P04.002	Coarse grit/ diamond particles	5,000	Edenta AG, Au SG, Switzerland
CeraGloss 3010 HP (Blue)	R09.003	Medium-coarse grit/ diamond particles	5,000	
CeraGloss 30010 HP (Yellow)	T02.001	Super-fine grit/ diamond particles	5,000	
LegabrilDiamond	08052307	diamond paste		Metalor Dental AG, Biel/Bienne, Switzerland

• RPM: Revolutions per minute

Fig. 7. Glaze material in paste form (IPS e.max Ceram Glaze, Ivoclar Vivadent AG, Schaan, Liechtenstein). The glaze paste was mixed with the IPS e.max Ceram Glaze and Stain Liquid until the desired consistency was reached (Experiment II).

Table 3. Glazing material used

Type	Brand name	Composition (%)	Lot No.	Manufacturer
Glazing paste	IPS e.max Ceram Glaze	SiO_2, 61.0 – 68.0 Al_2O_3, 5.0 – 8.0 Na_2O, 5.0 – 8.0 K_2O, 5.0 – 8.0 ZnO, 2.0 – 4.0 Other oxides, 3.5 – 17.0 Pigments, 0.0 – 1.0 Glycerine, 20.0 – 25.0 1,3-Butandiol, 15.0 – 20.0	R85911	Ivoclar Vivadent AG, Schaan, Liechtenstein

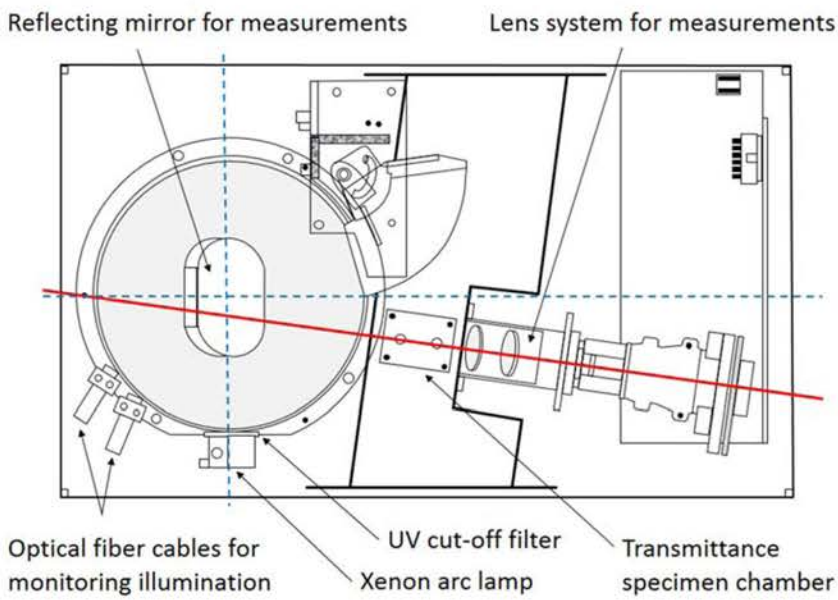

Fig. 8. A schematic view of reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan).

performing for the white calibration standard for reflectance measurements, and zero calibration box (CM-A 124, Minolta, Osaka, Japan) was for zero calibration for reflectance measurements. CIE L^*, a^* and b^* values were measured over a zero calibrating box (CM-A 124, Minolta, Osaka, Japan, CIE $L^* = 0.1099$, $a^* = 0.2107$ and $b^* = -0.4292$) with SCE under ultraviolet light excluded conditions and spectral reflectance over the white background (CM-A120, Minolta, Osaka, Japan, CIE $L^* = 96.6880$, $a^* = -0.1755$ and $b^* = -0.1236$) were measured in the range of visible wavelengths of 400 to 700 nm at 10 nm intervals. A drop of distilled water whose refractive index is 1.7, was placed between specimen and the background for better optical contact [28]. All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly.

Color coordinates, CIE L^*, a^* and b^*, were determined from the reflectance data using a computer software (Spectra-Magic version 1.01, Minolta, Osaka, Japan). Average L^*, a^* and b^* values were used to calculate CIE 1976 a,b (CIELAB) color difference, ΔE^*_{ab} of each group set using Eq. 1.

TP value of each subgroup within groups was calculated using Eq. 2. Spectral transmittance was recorded at 10 nm intervals in the range of 400 to 700 nm in the transmittance mode under ultraviolet light excluded conditions. The

aperture size was 9.56 mm in diameter which was made of an opaque black graphite plate with a central window, because the original aperture size of the instrument was 25 mm in diameter for the transmittance measurement. From the data, percent transmittance was calculated.

All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each subgroup in each group were obtained.

2.1.3. ***Experiment III****. Effect of the amount of thickness reduction on the color and translucency of monolithic zirconia*

One-hundred sixty-five square-shaped, pre-sintered zirconia blocks (20.0 mm × 20.0 mm × 2.7 mm) were prepared for Experiment III using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a brush (2850-B, Babara, Kobe, Japan). These specimens were divided into five groups according to the number of coloring liquid applications.

- Group I: One time of application
- Group II: Two times of application
- Group III: Three times of application

- Group IV: Four times of application
- Group V: Five times of application

All specimens were then sintered in a zirconia sintering furnace (Austromat *ba*SiC®, DEKEMA Dental-Keramiköfen GmbH, Freilassing, Germany). The sintering cycle was controlled as follows: The temperature was raised to 950°C for 63 minutes and maintained for 10 minutes, and then raised up to 1500°C for 55 minutes and maintained for 2 hours. After sintering process, the shrinkage of specimens was *circa* 20%. The mean size of sintered specimens was 16.3 mm × 16.3 mm, verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The specimen thicknesses were 1.997 ± 0.017 mm. Thirty-three specimens of each group were assigned to eleven subgroups according to the amount of thickness reduction (n = 3).

- Subgroup *-0 (Control group): No reduction
- Subgroup *-1: 0.1 mm reduction
- Subgroup *-2: 0.2 mm reduction
- Subgroup *-3: 0.3 mm reduction
- Subgroup *-4: 0.4 mm reduction
- Subgroup *-5: 0.5 mm reduction
- Subgroup *-6: 0.6 mm reduction
- Subgroup *-7: 0.7 mm reduction
- Subgroup *-8: 0.8 mm reduction
- Subgroup *-9: 0.9 mm reduction
- Subgroup *-10: 1.0 mm reduction

*: I, II, III, IV or V

Thickness reduction of each subgroup was performed on the colored surface using the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea) according to the protocol, respectively. Means and standard deviations of thickness of each subgroup are shown in Table 4. Colors were measured according to CIELAB color space relative to the standard illuminant D65 on a reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan), which was equipped with an integrating sphere. Illuminating and viewing configurations was followed by the protocol of

Table 4. Means and standard deviations in parentheses for the thickness of each subgroup

Subgroup	Thickness (mm)
0	2.00 (0.017)
1	1.91 (0.003)
2	1.81 (0.003)
3	1.71 (0.013)
4	1.61 (0.003)
5	1.51 (0.003)
6	1.41 (0.002)
7	1.31 (0.003)
8	1.21 (0.002)
9	1.11 (0.016)
10	1.01 (0.002)

Experiment II. Each of L^*, a^* and b^* values was measured over a zero calibrating box (CM-A 124, Minolta, Osaka, Japan, $L^* = 0.1099$, $a^* = 0.2107$ and $b^* = -0.4292$) and the white background (CM-A120, Minolta, Osaka, Japan, $L^* = 96.6880$, $a^* = -0.1755$ and $b^* = -0.1236$) in the reflectance mode with SCE under ultraviolet light excluded conditions at 10 nm intervals in the range of visible wavelengths of 400 to 700 nm. The aperture diameter of the reflectance measurement of the spectrophotometer (Target Mask CM-A 121, Minolta, Osaka, Japan) was 3 mm. Calibration of spectrophotometer was performed before measurements. A drop of distilled water was placed between specimen and the background for better optical contact. Spectral reflectance over the white background was also recorded at 10 nm intervals in the range of 400 to 700 nm.

Color coordinates, L^*, a^* and b^*, were determined from the reflectance data using a computer software (Spectra-Magic version 1.01, Minolta, Osaka, Japan). Average L^*, a^* and b^* values over the zero calibration box were used to calculate color difference, ΔE^*_{ab} of each group set using Eq. 1. TP value was obtained by calculating the color difference of the specimen over the black calibration box and white background using Eq. 2. Spectral transmittance was recorded at 10 nm intervals in the range of 400 to 700 nm in the transmittance mode under ultraviolet light excluded conditions. The aperture size was 9.56 mm in diameter which was made of an opaque black

graphite plate with a central window, because the original aperture size of the instrument was 25 mm in diameter for the transmittance measurement. From the data, percent transmittance was calculated.

All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each subgroup in each group were obtained.

2.2. Statistical analysis

SPSS software (version 20.0, SPSS Inc., Chicago, IL, USA) was used for statistical analyses and the probability level for statistical significance was set at $\alpha = 0.05$. One-way analysis of variance (ANOVA) and multiple comparison Scheffé test were performed to determine whether there were any significant differences in each parameter between the groups or subgroups.

For Experiment I, each of CIE L^*, a^*, b^* value, TP and OP value after coloring was used as a dependent variable, and the number of coloring liquid applications was used as an independent variable. The correlation between the number of coloring liquid applications and CIE L^*, a^*, b^* value, TP and OP value, was found out by using the Pearson correlation coefficient. The

linear regression was fitted to analyze the influences of the number of coloring liquid applications on CIE L^*, a^*, b^* value, TP and OP value.

For Experiment II, each of CIE L^*, a^*, b^* value and TP value was used as a dependent variable, and each surface treatment was used as an independent variable. Correlation between CIE L^*, a^* and b^* values of each surface treatment and the number of coloring liquid applications was found out by using the Pearson correlation coefficient. The linear regression was fitted to analyze the influences of the number of coloring liquid applications on CIE L^*, a^* and b^* values of each surface treatment.

For Experiment III, each of L^*, a^*, b^* value and TP value was used as a dependent variable, and the amount of thickness reduction was used as an independent variable. Correlation between the amount of thickness reduction and L^*, a^*, b^* value and TP value was found out by using Pearson correlation coefficient. The linear regression was fitted to analyze the influence of the amount of thickness reduction on L^*, a^*, b^* value and TP value.

3. RESULTS

3.1. Experiment I. Effect of the number of coloring liquid applications on the optical properties of monolithic zirconia

Spectral distributions and color coordinates were measured in the reflectance mode over the white and black backgrounds, and in the transmittance mode. Each spectral distribution in the different mode exhibited different spectral behavior (Fig. 9, 10 and 11). Means and standard deviations for CIE L^*, a^* and b^* values of each group are listed in Table 5. CIE L^* and b^* value were significantly influenced by the number of coloring liquid applications. An increase in the number of coloring liquid applications produced a decrease in the L^* value resulting in darker specimens and an increase in the b^* value resulting in more yellowish specimens, in the reflectance mode over a white and black background and in the transmittance mode. Fig. 12 showed changes in the CIE L^* and b^* values with the increase of the number of coloring liquid applications based on the reflected light over a white background. There was no significant difference in a^* value of reflectance mode over the white and black backgrounds among Group I, II and III. With regard to a^* value in transmittance mode, there was no significant difference among groups except for Group O (Table 5).

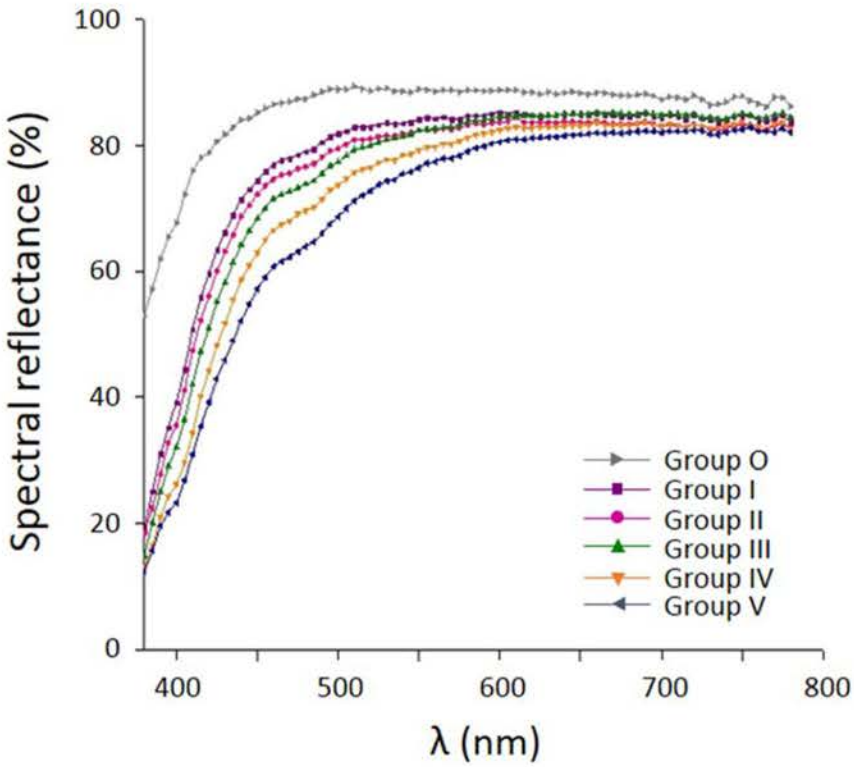

Fig. 9. Spectral reflectance over a white background of each group.

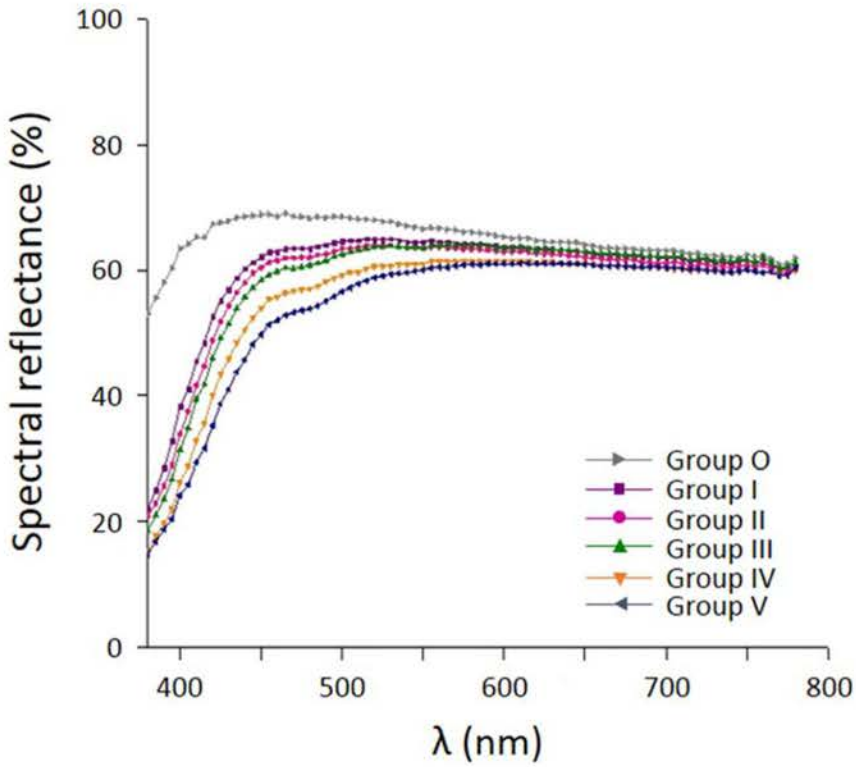

Fig. 10. Spectral reflectance over a black background of each group.

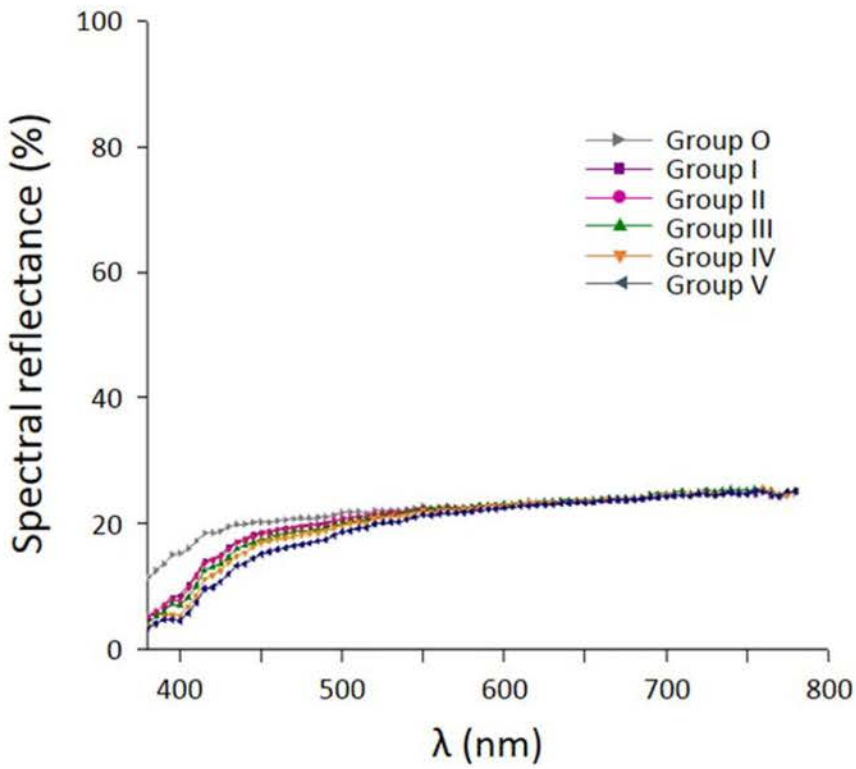

Fig. 11. Spectral transmittance of each group.

Table 5. Means (standard deviations) for CIE L^*, a^* and b^* values of each group

Group	Reflectance mode over the white background			Reflectance mode over the black background			Transmittance mode		
	L^*	a^*	b^*	L^*	a^*	b^*	L^*	a^*	b^*
O	95.38	-1.45[c]	2.95	85.52	-1.39	-1.37	54.22[a]	0.09	4.04
	(0.37)	(0.27)	(0.27)	(0.33)	(0.94)	(0.08)	(0.82)	(0.14)	(0.18)
I	93.17	-2.26[a]	8.36[a]	84.17	-2.38[a]	3.43[a]	53.76[a,b]	-0.37[a]	7.39[a]
	(0.61)	(0.12)	(1.03)	(0.34)	(0.08)	(0.97)	(0.53)	(0.15)	(0.77)
II	92.33[a]	-2.27[a]	9.35[a]	83.47[a]	-2.43[a,b]	4.10[a]	53.86[a,b]	-0.47[a]	7.80[a]
	(0.56)	(0.19)	(1.05)	(0.31)	(0.10)	(0.95)	(0.45)	(0.13)	(0.56)
III	92.14[a]	-2.10[a,b]	12.20	83.63[a]	-2.51[a,b]	6.26	53.69[a,b]	-0.29[a]	9.16
	(0.60)	(0.24)	(1.25)	(0.32)	(0.14)	(0.95)	(0.43)	(0.18)	(0.72)
IV	90.84	-1.98[b]	14.57	82.19	-2.60[c]	8.17	53.29[b]	-0.37[a]	10.31
	(0.39)	(0.20)	(0.79)	(0.17)	(0.15)	(0.39)	(0.34)	(0.17)	(0.54)
V	89.48	-1.49[c]	17.50	81.50	-2.55[b,c]	10.99	52.45	-0.28[a]	13.05
	(0.58)	(0.21)	(0.62)	(0.37)	(0.20)	(0.41)	(0.49)	(0.14)	(0.80)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

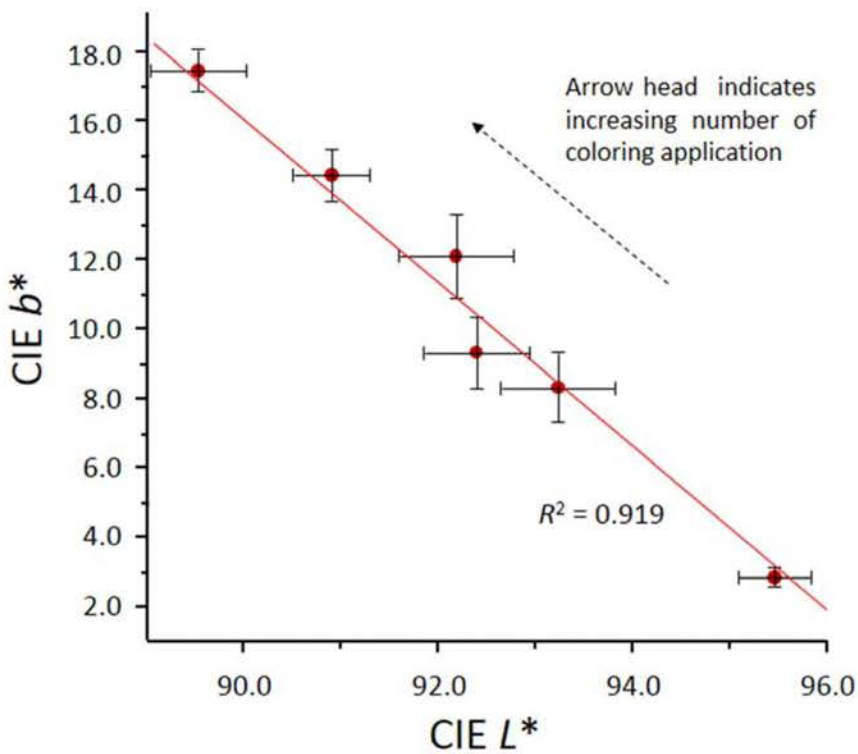

Fig. 12. Changes in the CIE L^* and b^* values with the increase of the number of coloring liquid applications based on the reflected light over a white background (Experiment I).

The color difference between each pair of groups was in the range from 1.3 to 15.7 ΔE^*_{ab} units (Table 6). The mean L^*, a^* and b^* values of each group over the white background in the reflectance mode were used to calculate the ΔE^*_{ab} between groups. The highest ΔE^*_{ab} value was 15.7 between Group O and V, while the lowest one was 1.3 between Group I and II. A perceptible color difference in a clinical setting ($\Delta E^*_{ab} > 3.7$) was obtained for all colored groups when compared with Group O. Color differences between two subsequent groups, such as I and II, II and III, III and IV, and IV and V, were not clinically perceptible ($\Delta E^*_{ab} < 3.7$).

Means and standard deviations of TP for each group are listed in Table 7. The statistical analyses showed no significant difference in TP value after the coloring procedure.

Means and standard deviations for the differences between the reflected colors over the black background and transmitted colors of each group are listed in Table 8. The OP values and Δb^* (the difference of CIE b^* value between transmitted color and reflected color) decreased as the number of coloring liquid applications increased. There was a negative correlation between the number of coloring liquid applications and OP value, indicating the Pearson correlation coefficients (r) to be −0.837. From a linear regression analysis, the coefficient of determination (R^2) was 0.701 (Fig. 13).

Table 6. Color differences between each group set

Group set	ΔE^*_{ab}
I-II	1.30
III-IV	2.71
II-III	2.85
IV-V	3.27
I-III	3.97
II-IV	5.43
O-I	5.90
III-V	5.96
I-IV	6.63
O-II	7.14
II-V	8.66
O-III	9.82
I-V	9.88
O-IV	12.48
O-V	15.70

• Group sets are arranged in ascending order of their ΔE^*_{ab} values.

Table 7. Means and standard deviations in parentheses for translucency parameter of each group

Group	TP
O	10.77[a] (0.47)
I	10.29[a] (0.41)
II	10.32[a] (0.41)
III	10.39[a] (0.43)
IV	10.80[a] (0.45)
V	10.39[a] (0.58)

- Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).
- TP denotes translucency parameter.

Table 8. Means and standard deviations in parentheses for the differences between the reflected and transmitted colors of each group

Group	OP	ΔE^*_{ab}	ΔL^*	Δa^*	Δb^*
O	5.61	31.81	31.31	1.48	5.41
	(0.20)	(1.08)	(1.08)	(0.11)	(0.20)
I	4.46[a]	30.74[a]	30.41[a]	2.01[a,b]	3.96[a]
	(0.54)	(0.56)	(0.55)	(0.16)	(0.64)
II	4.21[a,b]	29.91[a,b]	29.60[a,b]	1.95[a]	3.69[a,b]
	(0.71)	(0.75)	(0.69)	(0.17)	(0.87)
III	3.70[b,c]	30.17[a]	29.94[a]	2.22[b,c]	2.90[b,c]
	(0.57)	(0.33)	(0.32)	(0.18)	(0.81)
IV	3.14[c]	29.07[b]	28.89[b]	2.23[c]	2.14[c,d]
	(0.38)	(0.39)	(0.36)	(0.24)	(0.61)
V	3.11[c]	29.22[b]	29.05[b]	2.27[c]	2.06[d]
	(0.44)	(0.67)	(0.68)	(0.18)	(0.70)

- Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).
- OP denotes opalescence parameter.
- ΔE^*_{ab}, ΔL^*, Δa^* and Δb^* denote the differences of L^*, a^* and b^* values between transmitted and reflected color.

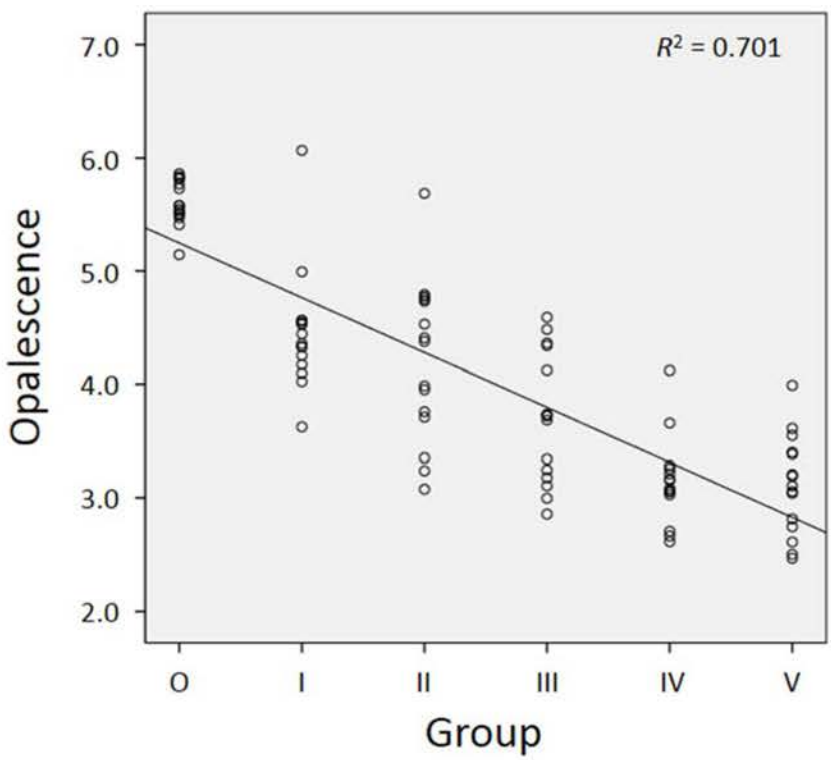

Fig. 13. Linear regression of opalescence values as a function of the number of coloring liquid applications (Experiment I).

Correlations between the number of coloring liquid applications and CIE L^*, a^* or b^* values were identified. There was a significant correlation between the number of coloring liquid applications and CIE L^* or b^* value indicating r value to be −0.937 or 0.968, R^2 to be 0.878 or 0.938, respectively (Fig. 14 and 15), whereas no significant correlation was found between the number of coloring liquid applications and CIE a^* value.

Correlations between OP and Δb^*, ΔE^*_{ab}, ΔL^* or Δa^* were identified. Between OP and Δb^*, r value was 0.991 and a regression equation, OP = $0.74\Delta b^* + 1.56$ ($R^2 = 0.982$) was calculated (Fig. 16). Based on the results of the present study, there were also significant correlations between OP and ΔE^*_{ab} ($r = 0.788$, $R^2 = 0.621$, Fig. 17), and OP and ΔL^* ($r = 0.736$, $R^2 = 0.541$, Fig. 18), but their correlations were lower than those between OP and Δb^*. However, there was a negative correlation between OP and Δa^* ($r = -0.782$, $R^2 = 0.612$, Fig. 19).

3.2. Experiment II. Effect of polishing and glazing on the color and translucency of monolithic zirconia

Means and standard deviations of L^*, a^* and b^* values over a zero calibrating box in the reflectance mode within each group as a function of surface treatment are listed in Table 9. As for L^* value, there was a significant difference between Subgroup N and P in each group, and between Subgroup

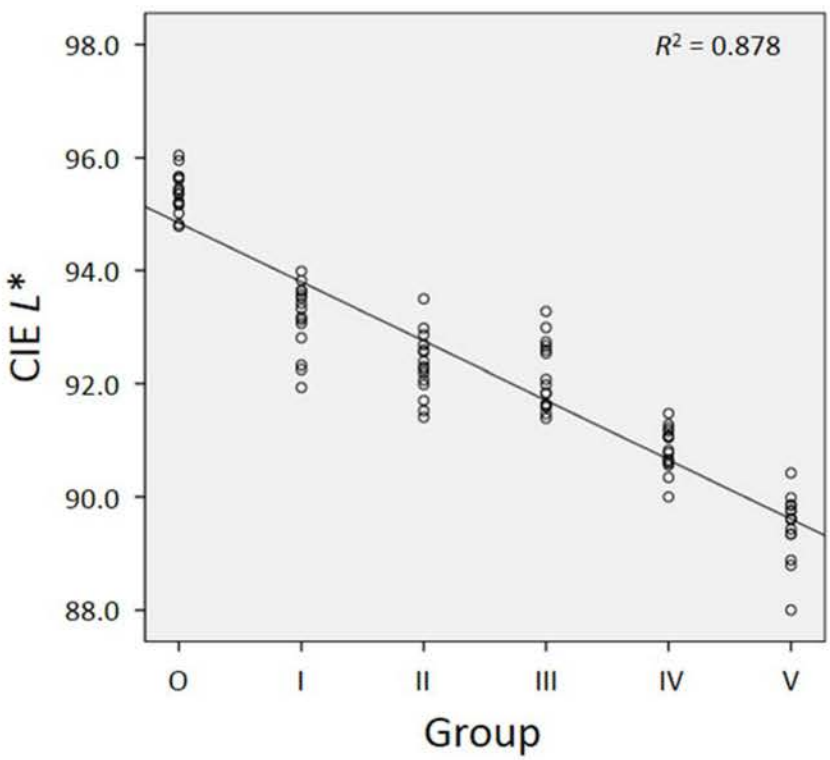

Fig. 14. Linear regression of CIE L^* values over a white background in the reflectance mode as a function of the number of coloring liquid applications (Experiment I).

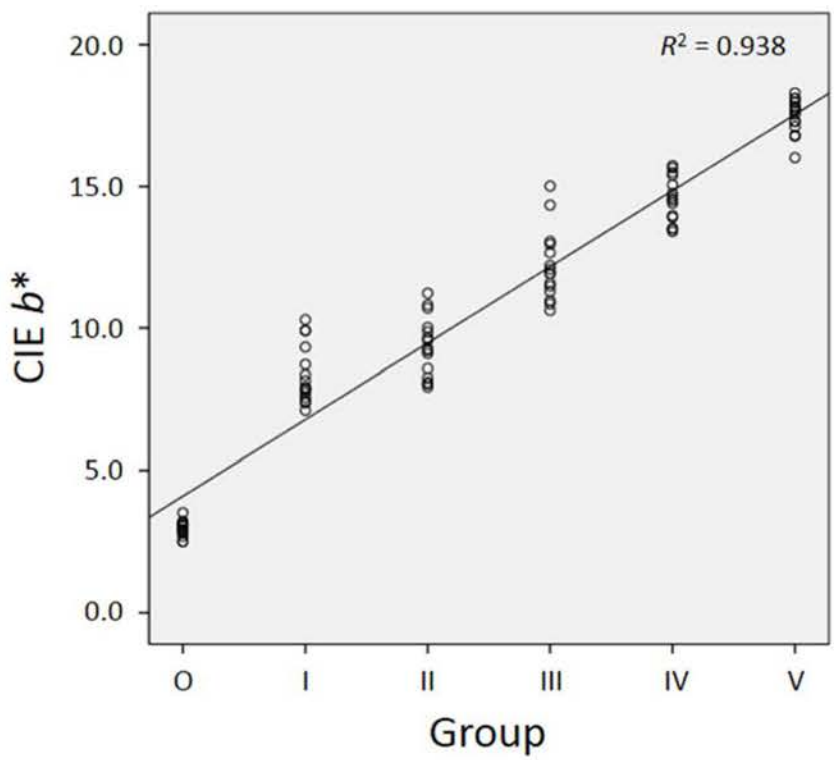

Fig. 15. Linear regression of CIE b^* values over a white background in the reflectance mode as a function of the number of coloring liquid applications (Experiment I).

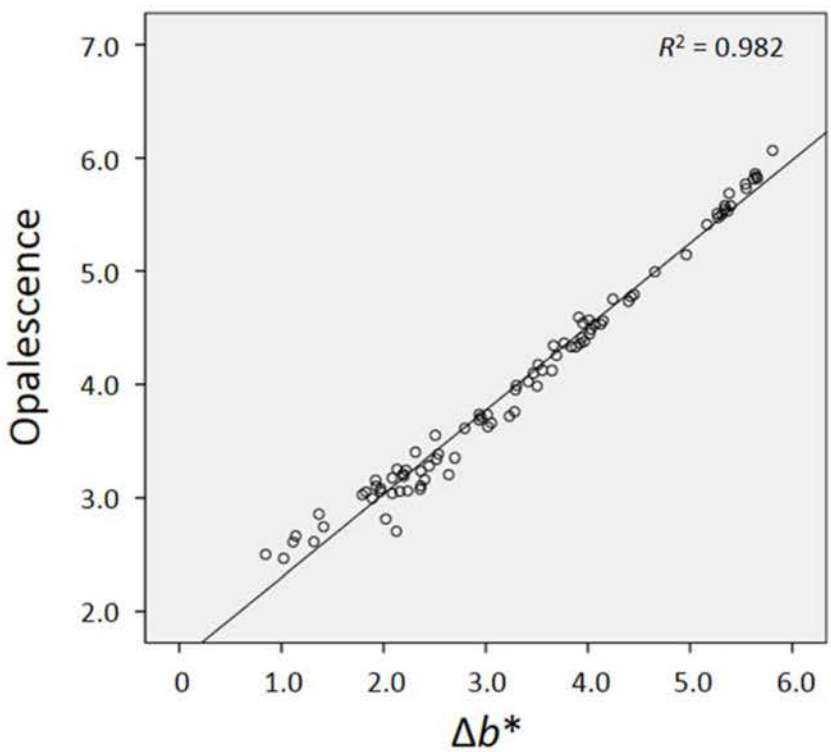

Fig. 16. Linear regression of opalescence values as a function of Δ*b** values (Experiment I).

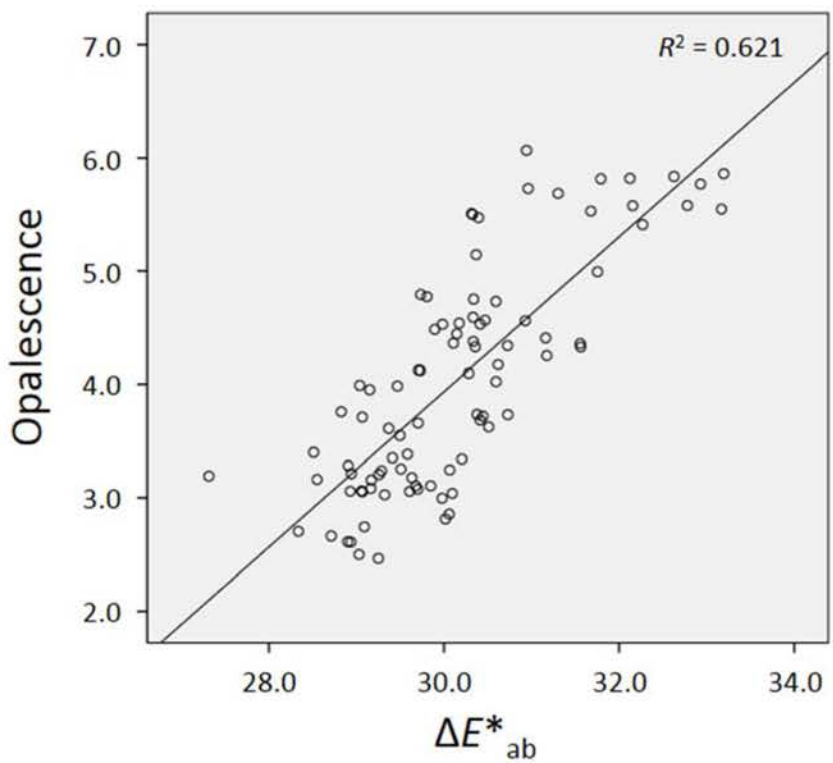

Fig. 17. Linear regression of opalescence values as a function of ΔE^*_{ab} values (Experiment I).

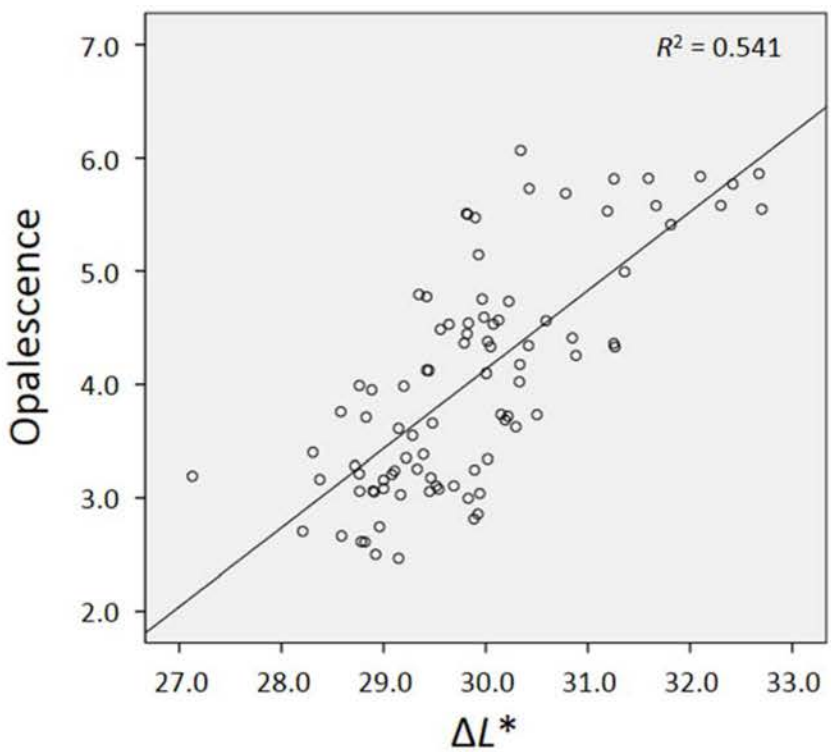

Fig. 18. Linear regression of opalescence values as a function of ΔL^* values (Experiment I).

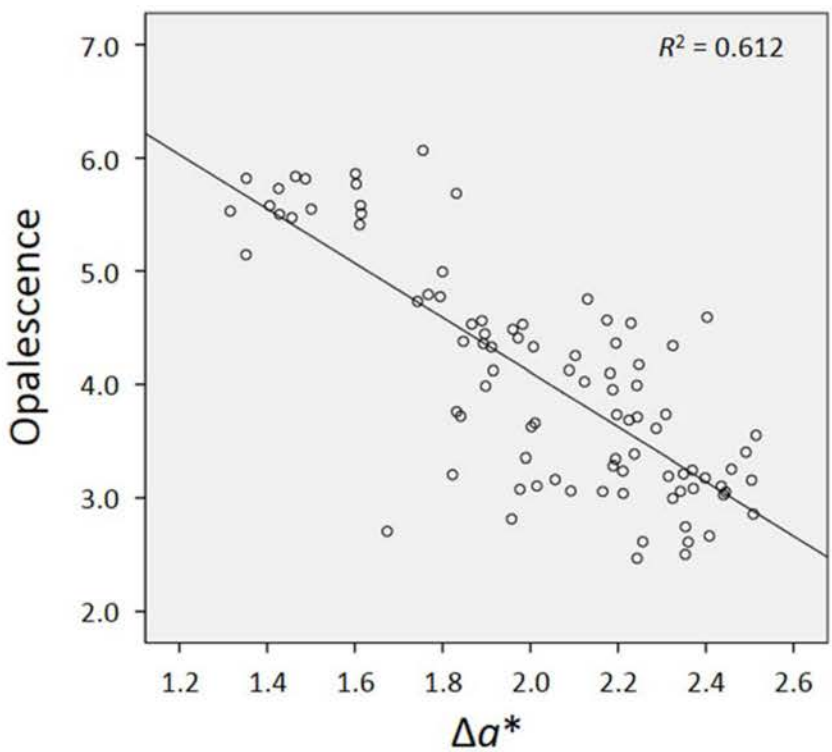

Fig. 19. Linear regression of opalescence values as a function of Δa^* values (Experiment I).

Table 9. Means (standard deviations) for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within each group as a function of surface treatment

	Surface treatment	Group				
		I	II	III	IV	V
L^*	N	73.49[a] (3.52)	70.49 (3.33)	70.92 (0.41)	71.17 (2.22)	66.37 (1.94)
	P	67.57[b] (3.82)	63.35 (2.47)	65.94[a] (2.87)	61.42 (1.45)	61.31[a] (3.07)
	G	70.61[a,b] (4.44)	67.04 (3.20)	66.97[a] (3.64)	64.58 (2.33)	61.27[a] (2.77)
a^*	N	-1.75[c] (0.14)	-2.12 (0.20)	-2.68[b] (0.11)	-2.33 (0.15)	-1.94[b] (0.28)
	P	-2.02[c,d] (0.29)	-2.66[a] (0.18)	-2.83[b,c] (0.19)	-3.07 (0.15)	-2.44 (0.33)
	G	-2.10[d] (0.45)	-2.67[a] (0.34)	-2.90[c] (0.21)	-2.90 (0.25)	-2.13[b] (0.28)
b^*	N	-2.87[e] (0.75)	-1.80[b] (0.68)	2.03 (0.73)	3.83[a] (0.73)	9.10[c] (1.02)
	P	-3.65[f] (0.24)	-1.71[b] (0.73)	3.47 (1.01)	3.43[a] (0.47)	9.80[c] (1.51)
	G	-2.72[e] (0.15)	-0.61 (0.70)	4.39 (0.70)	5.06 (0.87)	12.13 (0.70)

• Means with the same superscript letter in each group column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

N and G in each group except Group I. There was no significant difference between Subgroup P and G in Group I, III and V. As for a^* value, there was a significant difference between Subgroup N and P in Group II, IV and V and between Subgroup P and G in Group IV and V. There was a significant difference between Subgroup N and G except Group V. As for b^* value, there was no significant difference between Subgroup N and P in Group II, IV and V. There was a significant difference between Subgroup P and G in each group, and between Subgroup N and G in each group except Group I.

Fig. 20 to 27 show the spectral reflectance against the white background of specimens within groups or subgroups. Each surface treatment exhibited similar spectral behavior through the entire spectrum in the range of 400 to 700 nm, but the values of spectral reflectance in Subgroup P and G were generally lower than those in Subgroup N (Fig. 20 to 24). There was no significant difference between Subgroup P and G in Group I, III and V (Fig. 20, 22 and 24). There was a significant difference between each surface treatment in Group I, II and IV (Fig 20, 21 and 23), representing the highest value in Subgroup N and the lowest value in Subgroup P except for the short wavelengths of *circa* 400 nm ($\alpha = 0.05$). Fig. 25 to 27 presented the spectral reflectance of each group within Subgroup N, P and G. There was a gradually decreasing tendency of spectral reflectance as the number of coloring liquid

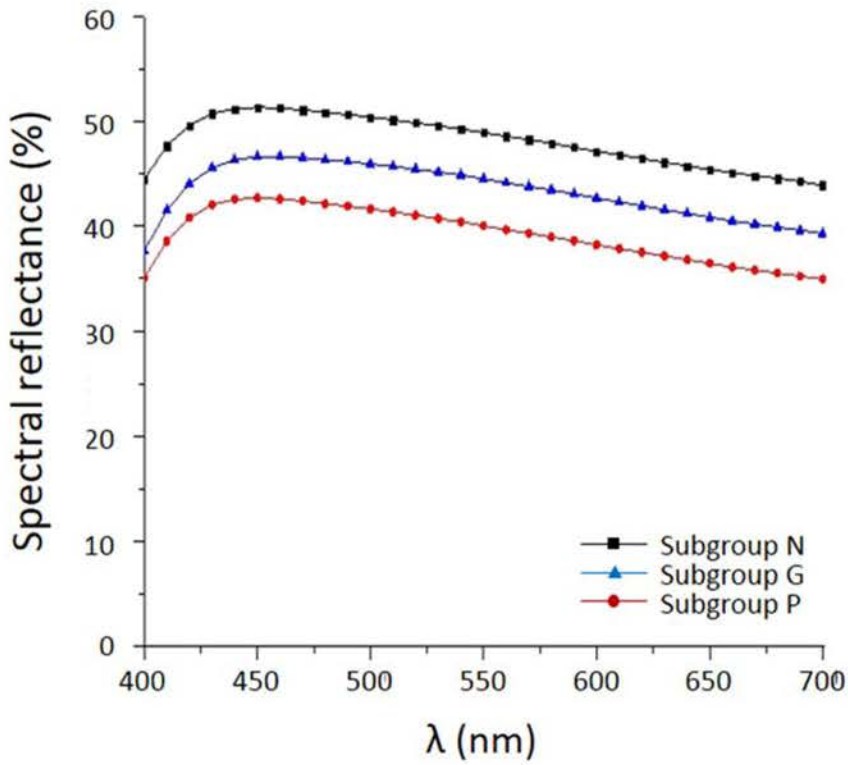

Fig. 20. Spectral reflectance of each subgroup in Group I against white background (Experiment II).

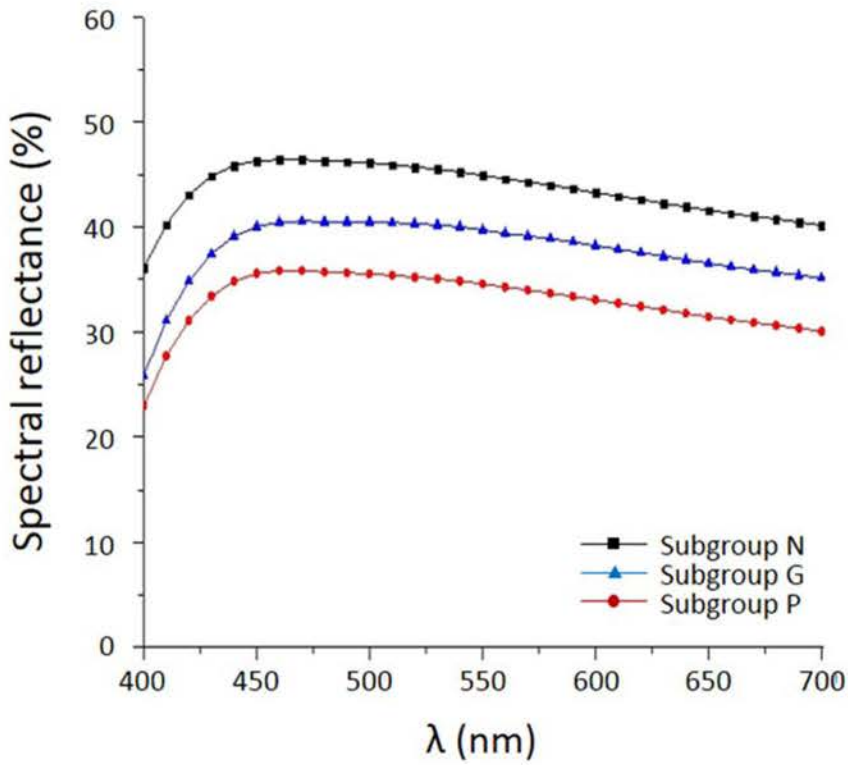

Fig. 21. Spectral reflectance of each subgroup in Group II against white background (Experiment II).

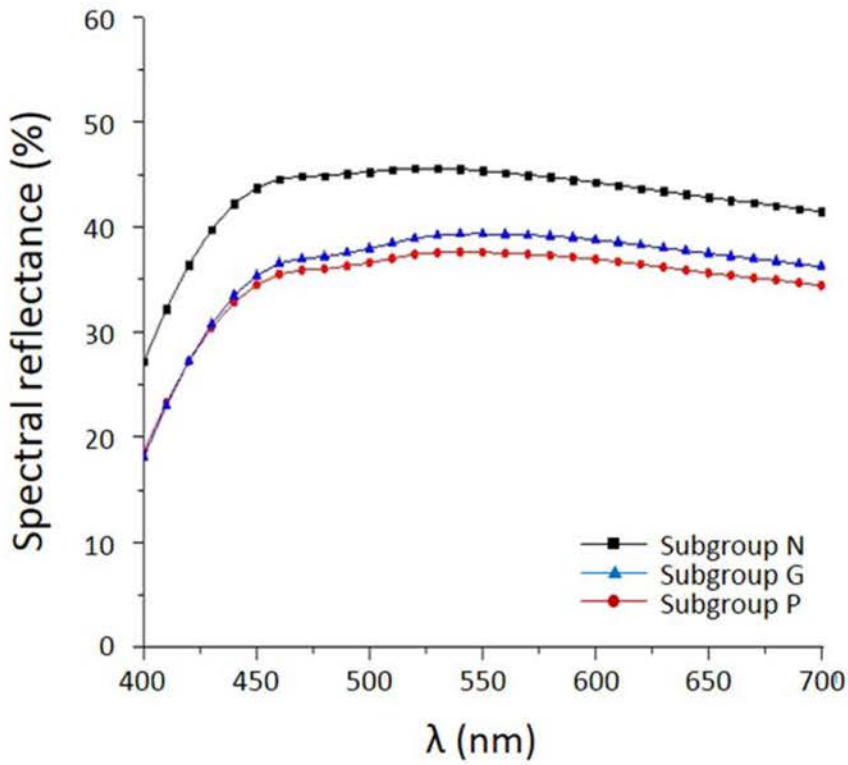

Fig. 22. Spectral reflectance of each subgroup in Group III against white background (Experiment II).

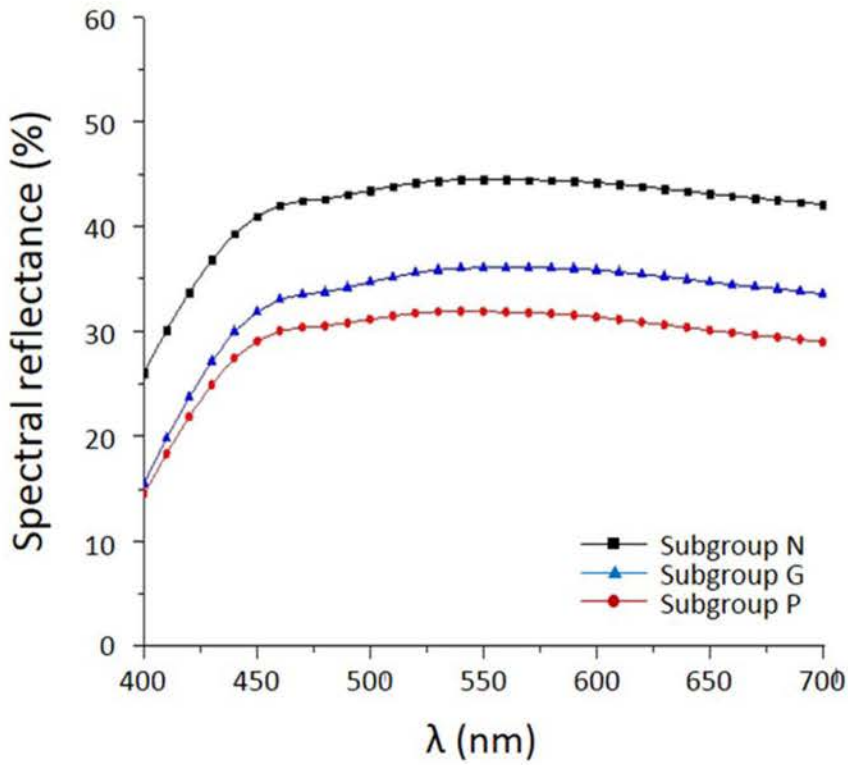

Fig. 23. Spectral reflectance of each subgroup in Group IV against white background (Experiment II).

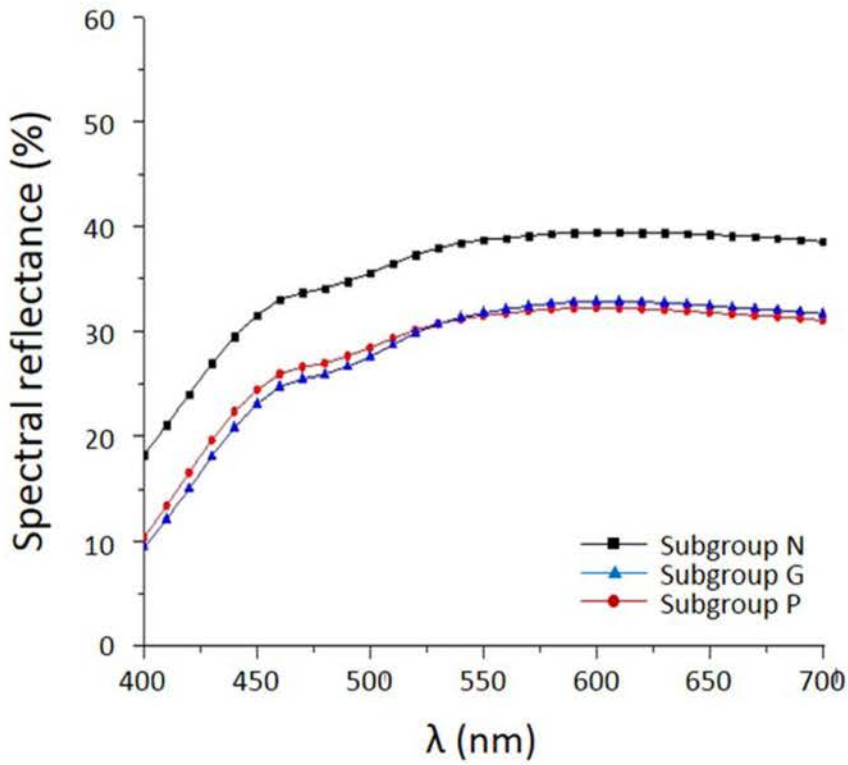

Fig. 24. Spectral reflectance of each subgroup in Group V against white background (Experiment II).

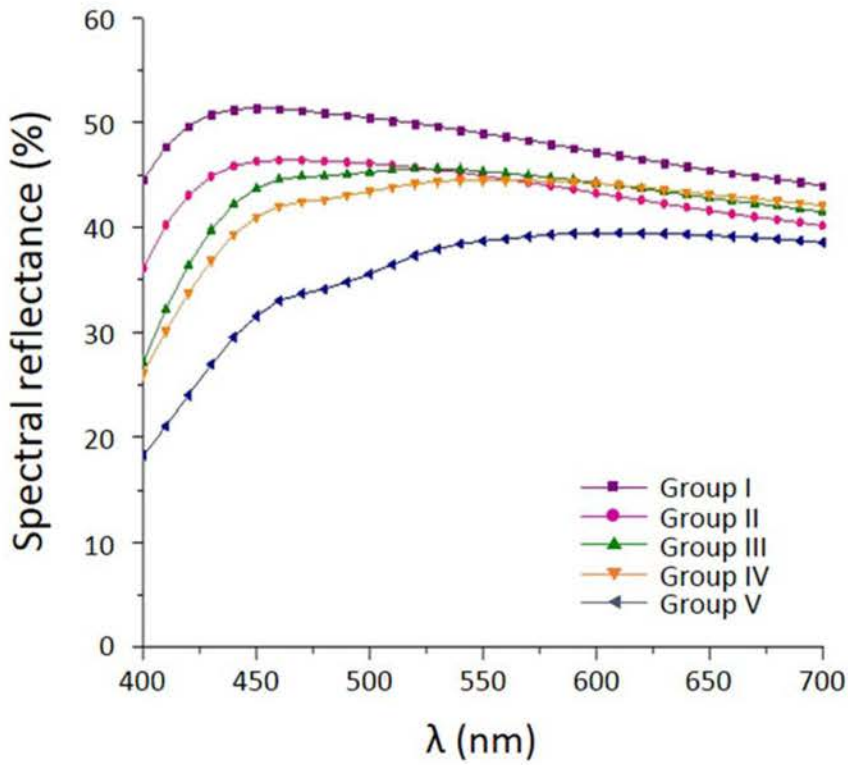

Fig. 25. Spectral reflectance of each group in Subgroup N (Experiment II).

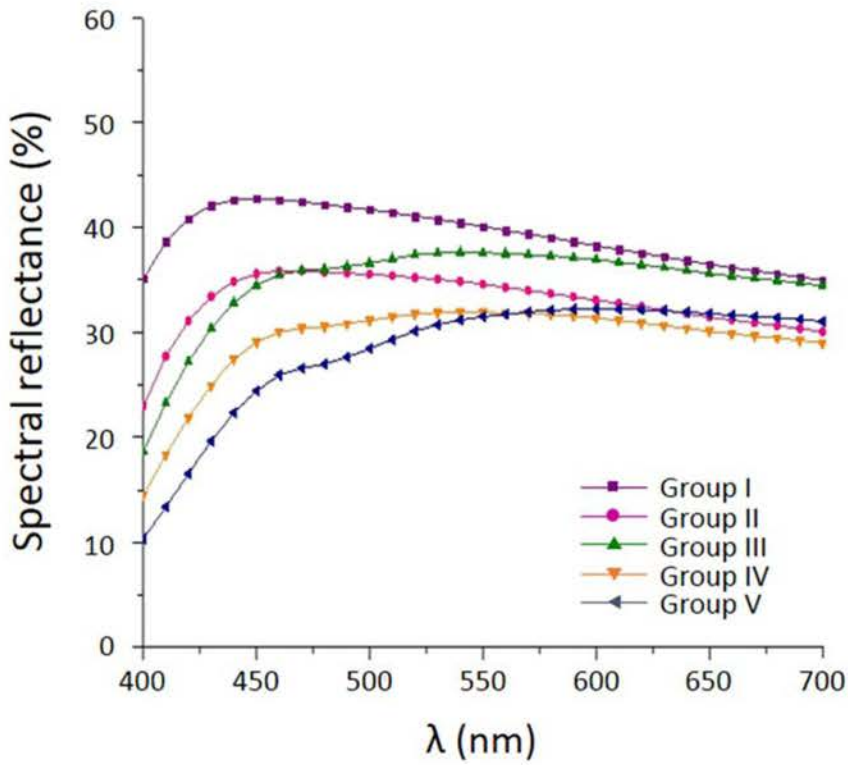

Fig. 26. Spectral reflectance of each group in Subgroup P (Experiment II).

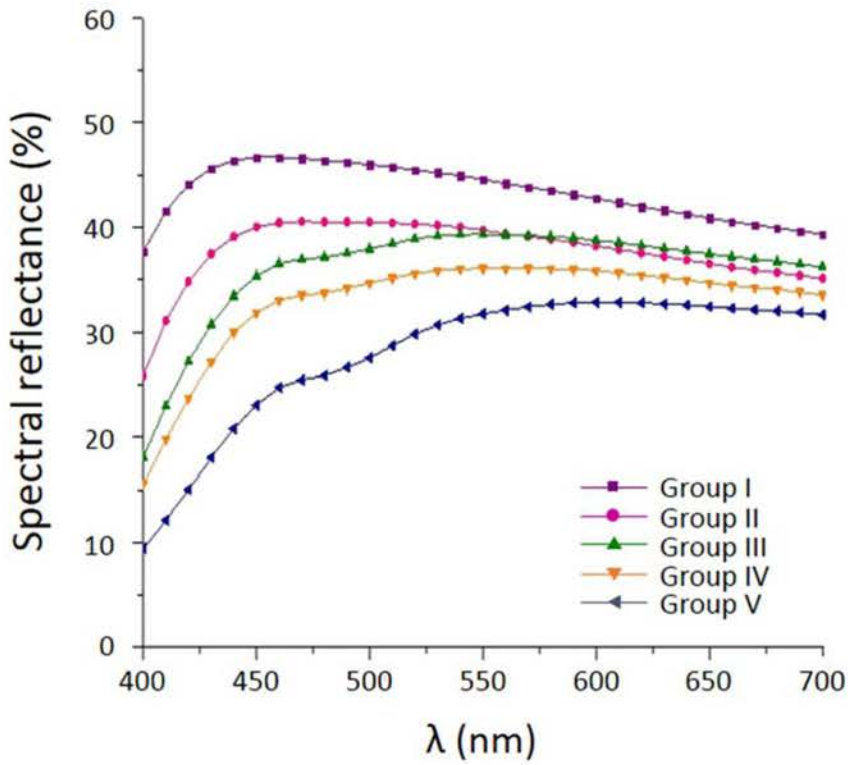

Fig. 27. Spectral reflectance of each group in Subgroup G (Experiment II).

applications increased in Subgroup N, P and G especially for the short wavelengths.

Color differences (ΔE^*_{ab}) between each pair of surface treatments within groups are shown in Table 10. Color difference between Subgroup N and P showed the highest values in comparison with the other pairs of surface treatments for the entire group ranged from 5.13 to 9.79 ΔE^*_{ab} units, which are clinically perceptible ($\Delta E^*_{ab} > 3.7$). Color difference between Subgroup N and G was in the range from 2.91 to 6.72 ΔE^*_{ab} units. A perceptible color difference was obtained between Subgroup N and G in Group III, IV and V. Color differences between Subgroup P and G are within the range of perceptibility threshold except Group II. Color differences between each group set in Subgroup N, P and G were shown in Table 11. Color difference between each pair of groups was in the range from 1.85 to 13.04 in Subgroup N, from 4.53 to 14.84 in Subgroup P, from 2.48 to 17.55 in Subgroup G, respectively. In general, a perceptible color difference was obtained in each group set.

Correlations between the number of coloring liquid applications and CIE L^*, a^* or b^* values in each surface treatment were identified. In all subgroups, CIE L^* tended to be decreased and CIE b^* value tended to be increased as the number of coloring liquid applications increased. There was a significant

Table 10. Color differences between each group set

Group	Subgroup set	ΔE^*_{ab}
I	N-P	5.98
	N-G	2.91
	P-G	3.17
II	N-P	7.17
	N-G	3.69
	P-G	3.85
III	N-P	5.19
	N-G	4.61
	P-G	1.38
IV	N-P	9.79
	N-G	6.72
	P-G	3.56
V	N-P	5.13
	N-G	5.93
	P-G	2.35

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

Table 11. Color differences between each group set

Subgroup	Group set	ΔE^*_{ab}
N	I-II	3.21
	I-III	5.61
	I-IV	7.12
	I-V	13.94
	II-III	3.89
	II-IV	5.68
	II-V	11.66
	III-IV	1.85
	III-V	8.45
	IV-V	7.14
P	I-II	4.70
	I-III	7.35
	I-IV	9.44
	I-V	14.84
	II-III	5.80
	II-IV	5.51
	II-V	11.69
	III-IV	4.53
	III-V	7.85
	IV-V	6.40
G	I-II	4.19
	I-III	8.03
	I-IV	9.87
	I-V	17.55
	II-III	5.01
	II-IV	6.18
	II-V	14.00
	III-IV	2.48
	III-V	9.64
	IV-V	7.85

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

correlation between the number of coloring liquid applications and CIE b^* value indicating r value to be 0.960 and R^2 to be 0.922 in Subgroup N, r value to be 0.948 and R^2 to be 0.899 in Subgroup P, and r value to be 0.962 and R^2 to be 0.925 in Subgroup G, respectively (Fig. 28). There was a negative correlation between the number of coloring liquid applications and CIE L^* value in each surface treatment (Fig. 29), whereas no significant correlation was found between the number of coloring liquid applications and CIE a^* value (Fig. 30).

Means and standard deviations for TP values within each group as a function of surface treatment are listed in Table 12. The statistical analyses showed no significant difference in TP values between each subgroup in Group I, II and II. There was no significant difference in TP values between Subgroup P and G in Group IV and V (Fig. 31).

Fig. 32 to 39 show the spectral transmittance behavior within groups or subgroups. There was no distinct difference between each subgroup in all groups. Fig. 37 shows that there was a decrease in spectral transmittance value with the increase of number of coloring liquid applications in Subgroup N and this tendency corresponds to Subgroup P and G (Fig. 38 and 39).

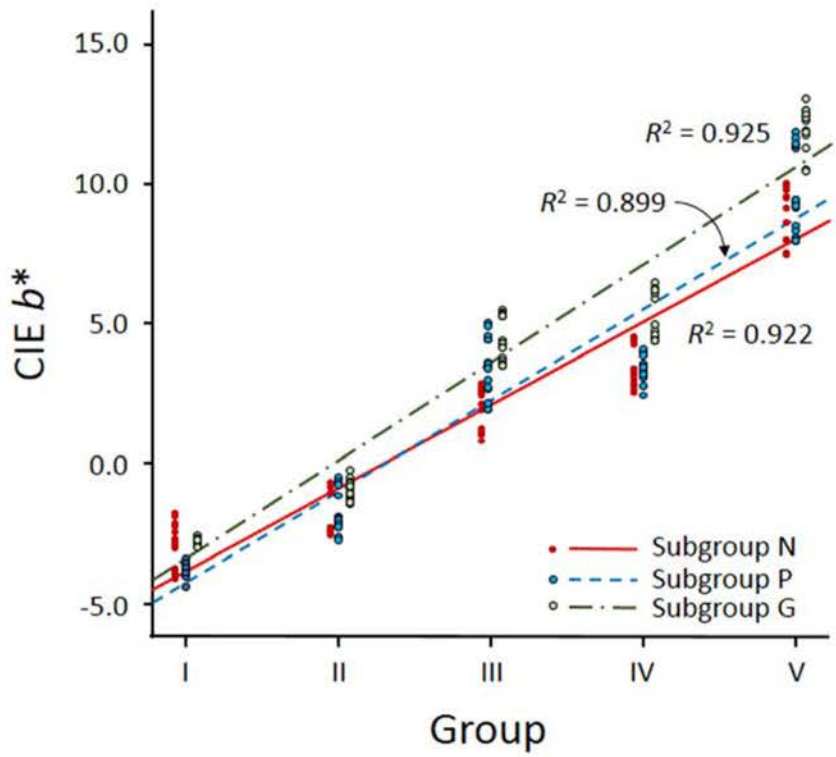

Fig. 28. Linear regression of CIE b^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

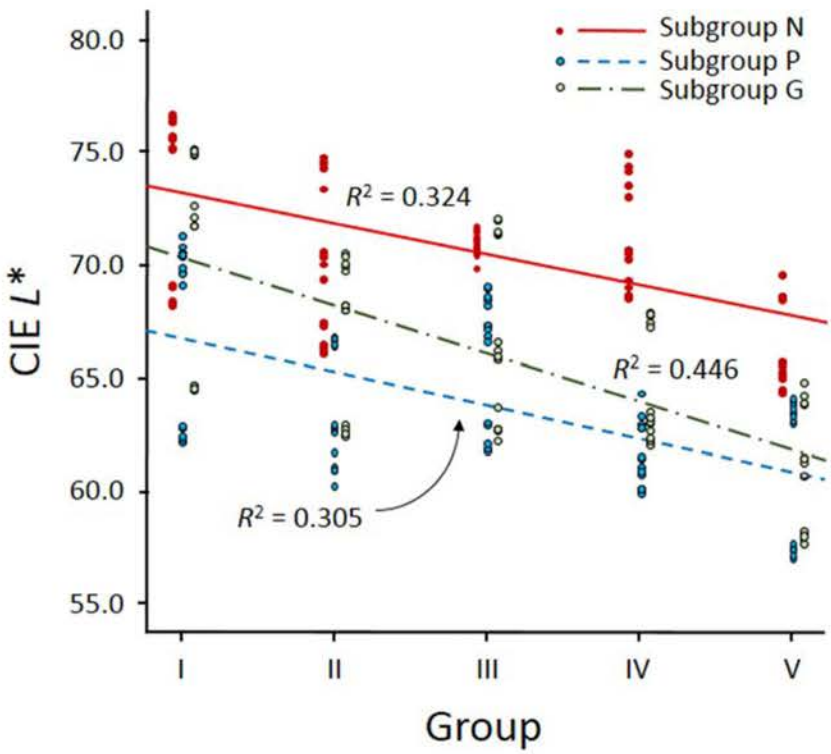

Fig. 29. Linear regression of CIE L^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

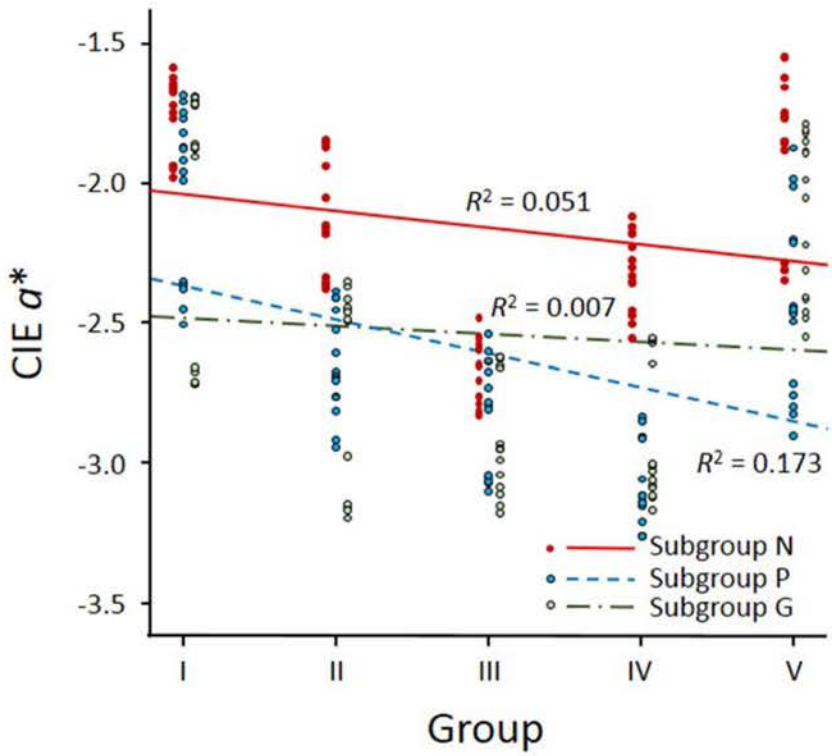

Fig. 30. Linear regression of CIE a^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

Table 12. Means and standard deviations in parentheses for translucency parameter of each group as a function of surface treatment

	Surface treatment	Group				
		I	II	III	IV	V
TP	N	1.87[a] (0.48)	2.02[a] (0.41)	2.21[a] (0.34)	2.05[a] (0.42)	1.74[a] (0.41)
	P	1.96[a] (0.52)	1.85[a] (0.32)	2.13[a] (0.17)	2.19[a] (0.24)	1.85[a,b] (0.31)
	G	1.85[a] (0.24)	2.00[a] (0.46)	2.16[a] (0.41)	2.18[a] (0.28)	2.06[b] (0.17)

• Means with the same superscript letter in each group column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

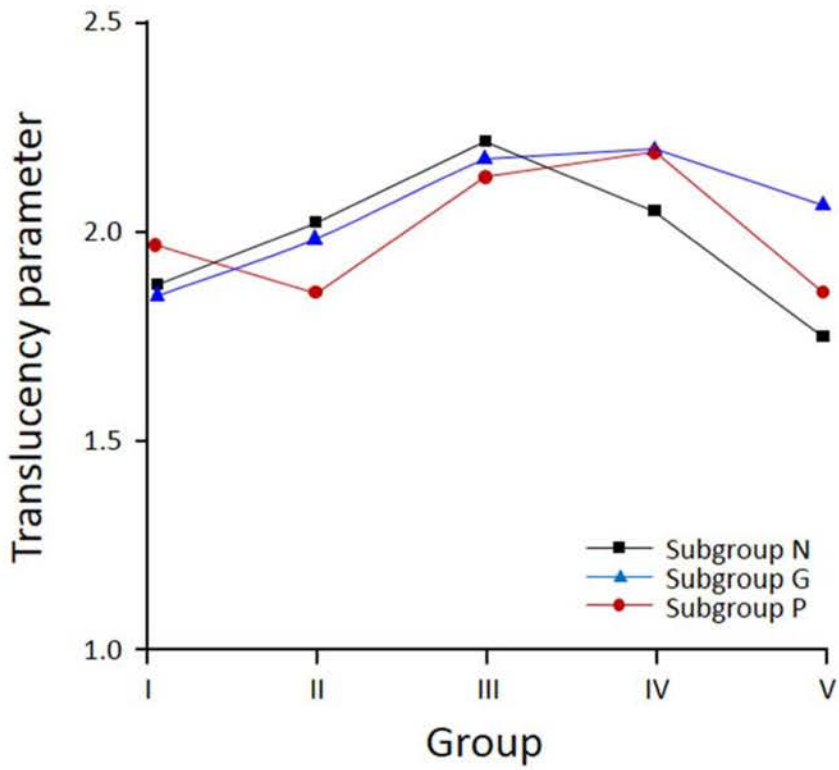

Fig. 31. Means of translucency parameter values of each subgroup as a function of the number of coloring liquid applications (Experiment II).

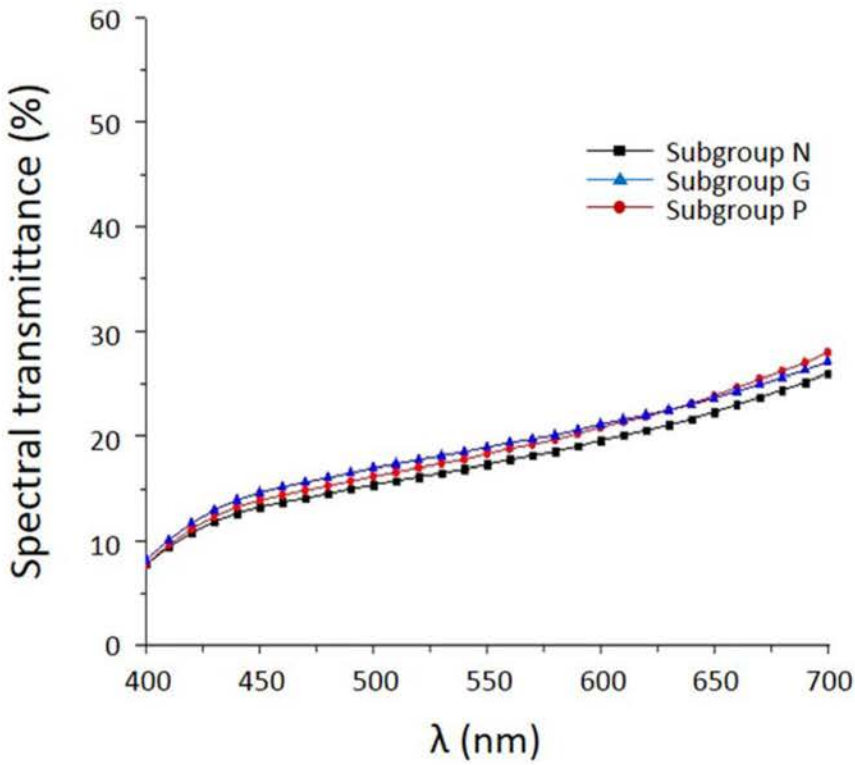

Fig. 32. Spectral transmittance of each subgroup in Group I (Experiment II).

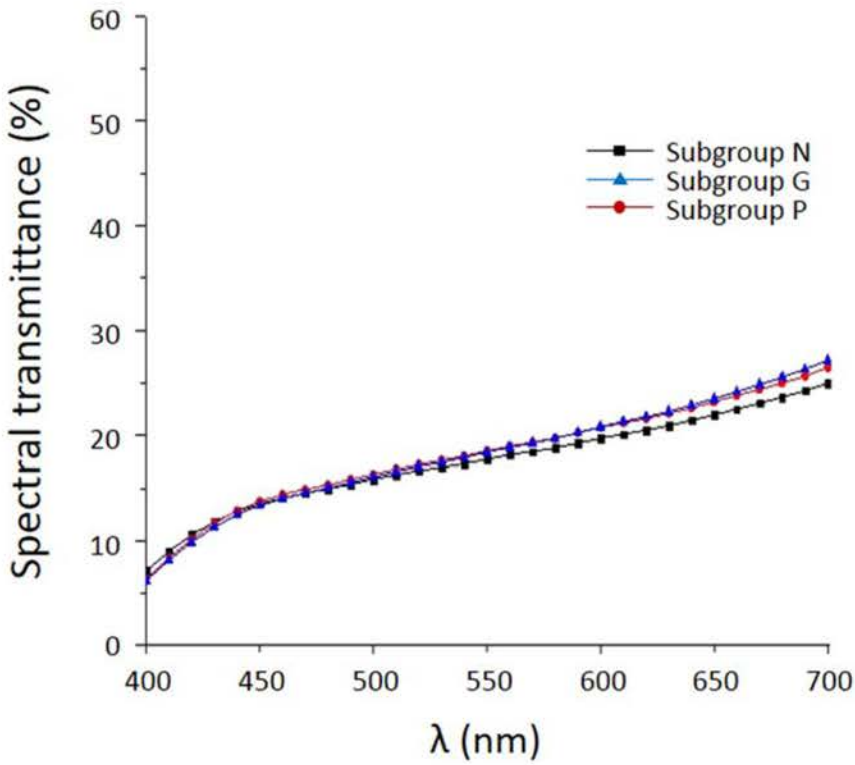

Fig. 33. Spectral transmittance of each subgroup in Group II (Experiment II).

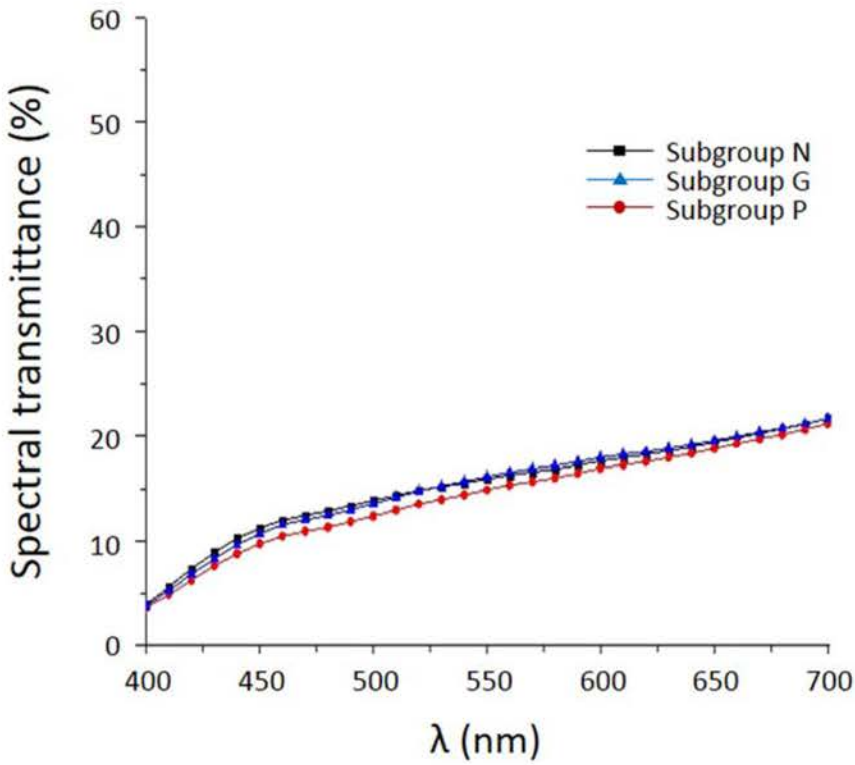

Fig. 34. Spectral transmittance of each subgroup in Group III (Experiment II).

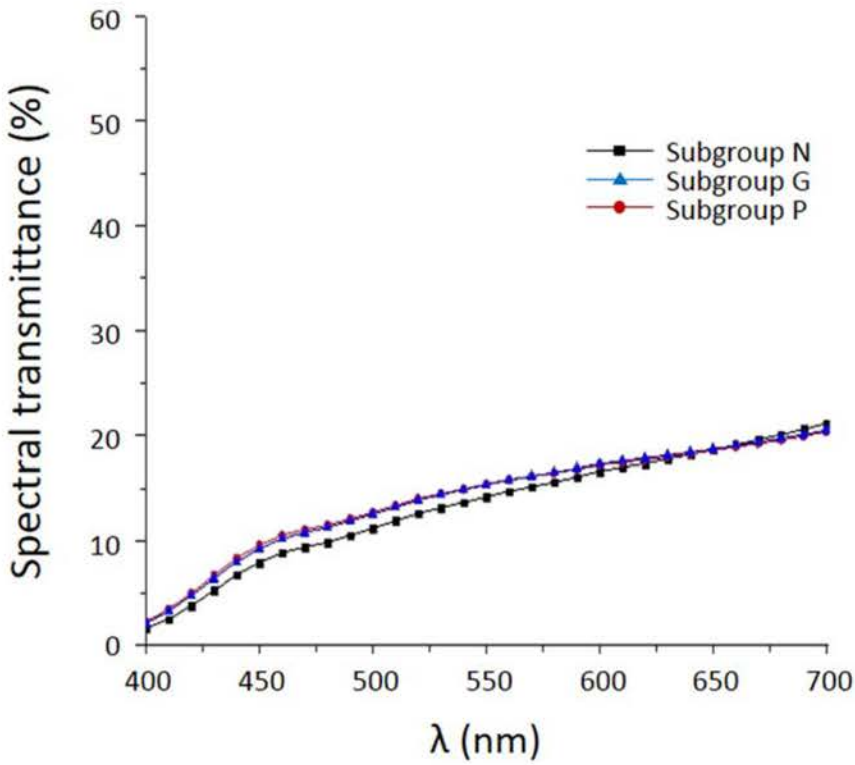

Fig. 35. Spectral transmittance of each subgroup in Group IV (Experiment II).

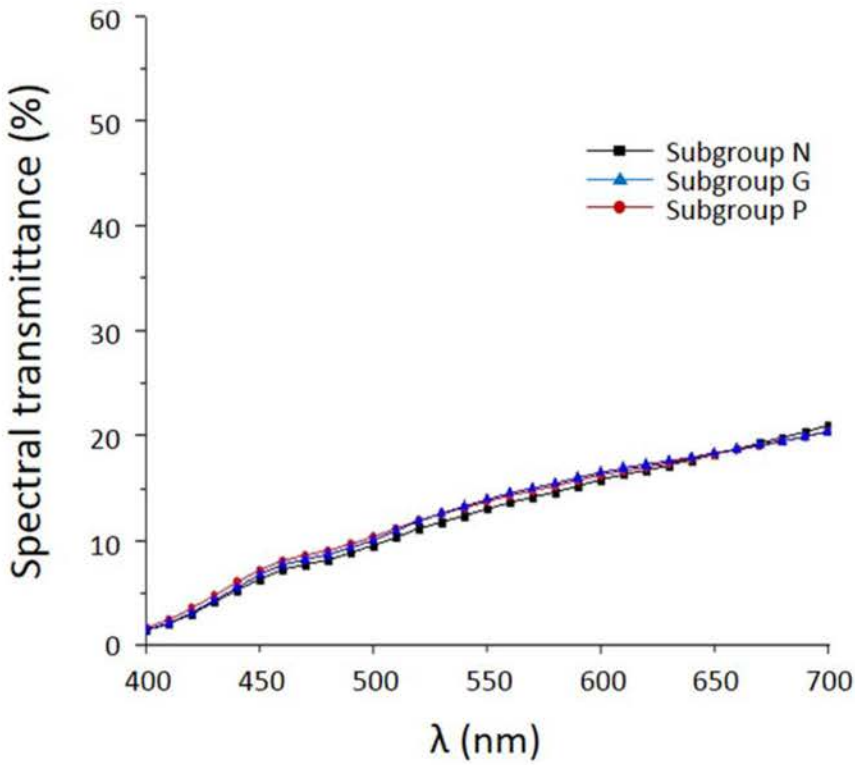

Fig. 36. Spectral transmittance of each subgroup in Group V (Experiment II).

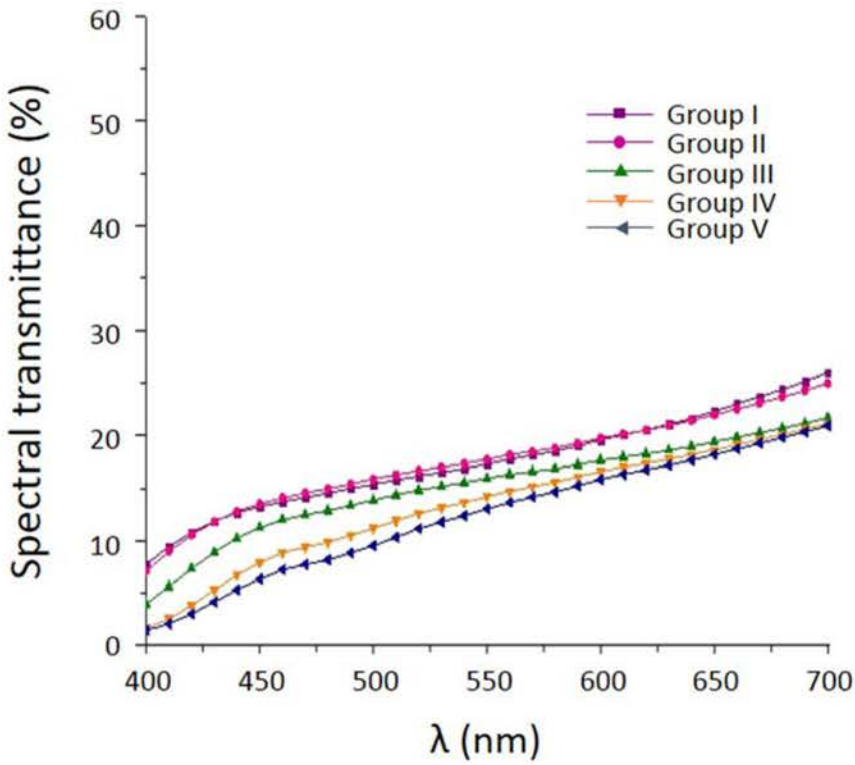

Fig. 37. Spectral transmittance of each group in Subgroup N (Experiment II).

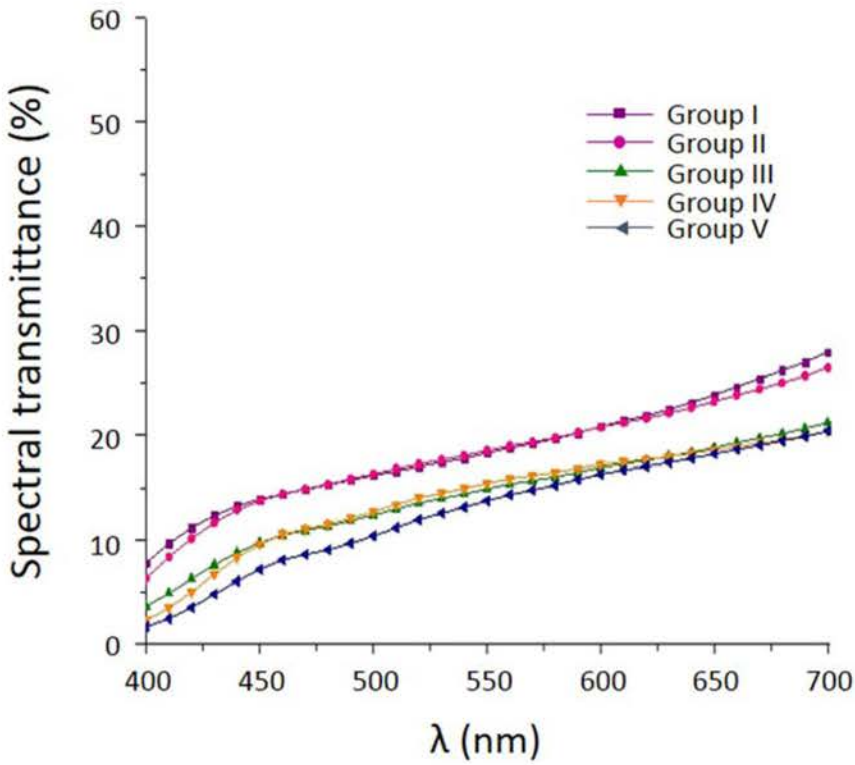

Fig. 38. Spectral transmittance of each group in Subgroup P (Experiment II).

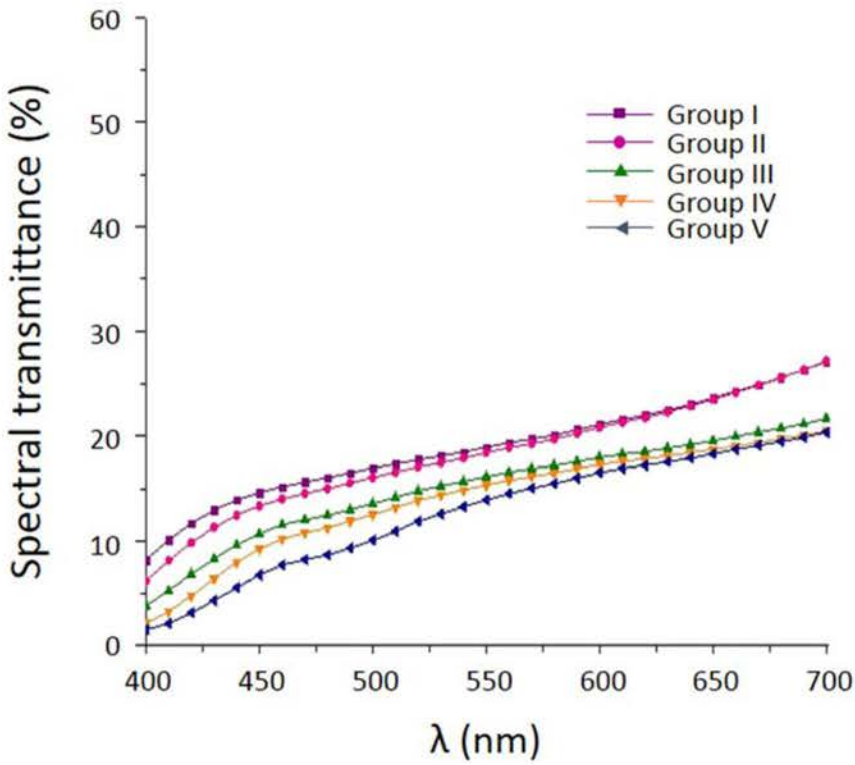

Fig. 39. Spectral transmittance of each group in Subgroup G (Experiment II).

3.3. Experiment III. Effect of the amount of thickness reduction on the color and translucency of monolithic zirconia

Means and standard deviations of L^*, a^* and b^* values over a zero calibrating box in the reflectance mode within each group as a function of the amount of thickness reduction are listed in Table 13-1 to 13-5. In Group I (Table 13-1), there was a significant difference between Subgroup 0 and other subgroups for L^*, and b^* value. In Group II (Table 13-2), there was a significant difference among Subgroup 0, Subgroup 1 and other subgroups for L^* value, and b^* value. In Group III (Table 13-3), there was a significant difference between Subgroup 0 and other subgroups for L^* value and there was a significant difference among Subgroup 0, Subgroup 1 and other subgroups for b^* value. In Group IV (Table 13-4), there was a significant difference between Subgroup 0 and other subgroups for L^* value, and b^* value generally decreased. In Group V (Table 13-5), there was a significant difference between Subgroup 0 and other subgroups for L^* value, and b^* value generally decreased. On the contrary, a^* values generally increased as the amount of thickness reduction increased in all groups.

Correlation between L^*, a^* or b^* value and the amount of thickness reduction is presented in Fig. 40 to 42. There were negative, but weak correlations between L^* value and the amount of thickness reduction in Group I, II and III (Fig. 40). There were positive correlations between a^* value and the amount

Table 13-1. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group I as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
I	0	75.20 (1.96)	$-1.74^{a,b}$ (0.13)	-2.93 (0.51)
	1	68.19^{b} (0.79)	-1.78^{a} (0.04)	-4.86^{a} (0.26)
	2	68.41^{b} (0.82)	$-1.60^{c,d,e}$ (0.05)	$-4.57^{a,b}$ (0.11)
	3	$67.72^{a,b}$ (0.43)	$-1.55^{d,e,f}$ (0.07)	-4.74^{a} (0.12)
	4	66.65^{a} (0.28)	$-1.62^{c,d}$ (0.03)	-4.86^{a} (0.15)
	5	$67.10^{a,b}$ (0.40)	$-1.67^{b,c}$ (0.04)	-4.69^{a} (0.18)
	6	$67.24^{a,b}$ (0.55)	$-1.59^{d,e}$ (0.03)	$-4.60^{a,b}$ (0.14)
	7	66.44^{a} (0.29)	$-1.62^{c,d}$ (0.05)	$-4.59^{a,b}$ (0.08)
	8	$67.41^{a,b}$ (0.71)	$-1.51^{e,f}$ (0.07)	$-4.27^{b,c}$ (0.21)
	9	68.15^{b} (0.51)	$-1.46^{f,g}$ (0.05)	-4.13^{c} (0.14)
	10	67.76^{b} (1.06)	-1.40^{g} (0.08)	-4.08^{c} (0.24)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-2. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group II as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
II	0	75.35 (2.78)	-2.38 (0.19)	0.35 (0.20)
	1	73.25 (2.86)	-1.72^{a} (0.09)	-3.04 (0.82)
	2	68.11^{a} (0.15)	$-1.62^{a,b}$ (0.05)	$-4.34^{b,c}$ (0.05)
	3	66.99^{a} (0.52)	$-1.62^{a,b}$ (0.06)	-4.85^{a} (0.17)
	4	67.02^{a} (0.15)	$-1.61^{a,b}$ (0.03)	$-4.69^{a,b}$ (0.08)
	5	67.33^{a} (0.26)	$-1.64^{a,b}$ (0.03)	$-4.54^{a,b,c}$ (0.04)
	6	67.67^{a} (0.54)	$-1.60^{a,b}$ (0.04)	$-4.50^{a,b,c}$ (0.21)
	7	67.42^{a} (0.25)	$-1.59^{a,b}$ (0.04)	$-4.33^{b,c}$ (0.05)
	8	67.20^{a} (0.33)	$-1.55^{b,c}$ (0.03)	$-4.38^{b,c}$ (0.11)
	9	68.01^{a} (0.13)	-1.44^{c} (0.60)	-4.22^{c} (0.07)
	10	67.87^{a} (0.19)	-1.29 (0.16)	-4.16^{c} (0.06)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-3. Means and standard deviations in parentheses for CIE *L**, *a** and *b** values over a zero calibration box in the reflectance mode within Group III as a function of the amount of thickness reduction

Group	Subgroup	*L**	*a**	*b**
III	0	72.67 (1.88)	-2.72 (0.10)	3.34 (0.88)
	1	67.51[b] (1.10)	-2.32 (0.17)	-2.04 (0.71)
	2	67.22[a,b] (0.45)	-1.83 (0.07)	-3.80[c] (0.11)
	3	67.33[a,b] (0.35)	-1.61[a,b] (0.05)	-4.62[a.b] (0.16)
	4	66.28[a] (0.34)	-1.63[a] (0.03)	-4.86[a] (0.13)
	5	67.45[a,b] (0.28)	-1.66[a] (0.02)	-4.52[a,b] (0.06)
	6	67.71[b] (0.75)	-1.55[a,b] (0.04)	-4.46[a,b] (0.19)
	7	67.03[a,b] (0.45)	-1.61[a] (0.04)	-4.40[a,b] (0.10)
	8	67.62[b] (0.20)	-1.50[b,c] (0.04)	-4.25[b,c] (0.09)
	9	68.23[b] (0.35)	-1.41[c,d] (0.06)	-4.20[b,c] (0.09)
	10	67.97[b] (0.22)	-1.37[d] (0.04)	-4.13[b,c] (0.11)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-4. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group IV as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
IV	0	72.70 (1.19)	-2.21^{b} (0.11)	5.79^{a} (0.33)
	1	64.38^{a} (1.44)	-2.52^{a} (0.14)	4.85^{a} (0.50)
	2	$65.42^{a,b}$ (0.55)	-2.62^{a} (0.10)	$1.45^{b,c}$ (1.65)
	3	65.69^{b} (0.79)	-2.56^{a} (0.12)	1.60^{b} (1.94)
	4	66.04^{b} (1.18)	-2.20^{b} (0.35)	-0.76^{c} (3.62)
	5	67.43^{c} (0.40)	-1.89^{c} (0.24)	-3.34^{d} (1.34)
	6	67.55^{c} (0.26)	-1.62^{d} (0.03)	-4.43^{d} (0.10)
	7	67.43^{c} (0.62)	$-1.66^{c,d}$ (0.12)	-4.15^{d} (0.44)
	8	67.61^{c} (0.25)	-1.55^{d} (0.04)	-4.25^{d} (0.10)
	9	68.22^{c} (0.12)	-1.46^{d} (0.04)	-4.13^{d} (0.12)
	10	67.89^{c} (0.29)	-1.43^{d} (0.04)	-4.09^{d} (0.10)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-5. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group V as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
V	0	68.97 (2.91)	$-1.85^{b,c}$ (0.56)	9.01 (2.36)
	1	64.29^{a} (1.39)	-2.65^{a} (0.05)	4.66 (0.52)
	2	$65.47^{a,b}$ (0.59)	-2.60^{a} (0.05)	3.01 (1.04)
	3	$66.91^{b,c}$ (0.89)	-2.10^{b} (0.15)	-2.40 (0.35)
	4	67.33^{c} (0.59)	$-1.77^{c,d}$ (0.07)	-3.81^{a} (0.14)
	5	67.40^{c} (0.28)	$-1.70^{c,d,e}$ (0.05)	-4.26^{a} (0.21)
	6	67.73^{c} (0.18)	$-1.60^{c,d,e,f}$ (0.04)	-4.34^{a} (0.06)
	7	$66.97^{b,c}$ (0.16)	$-1.58^{c,d,e,f}$ (0.05)	-4.51^{a} (0.06)
	8	67.72^{c} (0.14)	$-1.50^{d,e,f}$ (0.07)	-4.36^{a} (0.07)
	9	68.90 (0.70)	-1.41^{f} (0.06)	-4.00^{a} (0.22)
	10	67.76^{c} (0.54)	$-1.42^{e,f}$ (0.05)	-4.06^{a} (0.15)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

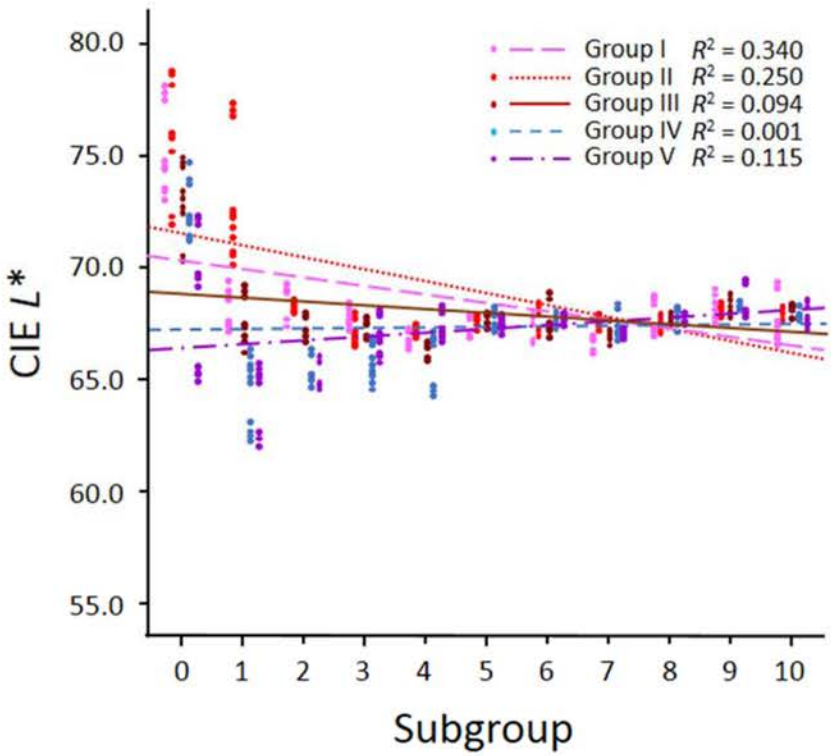

Fig. 40. Linear regression of CIE L^* values of each group as a function of the amount of thickness reduction (Experiment III).

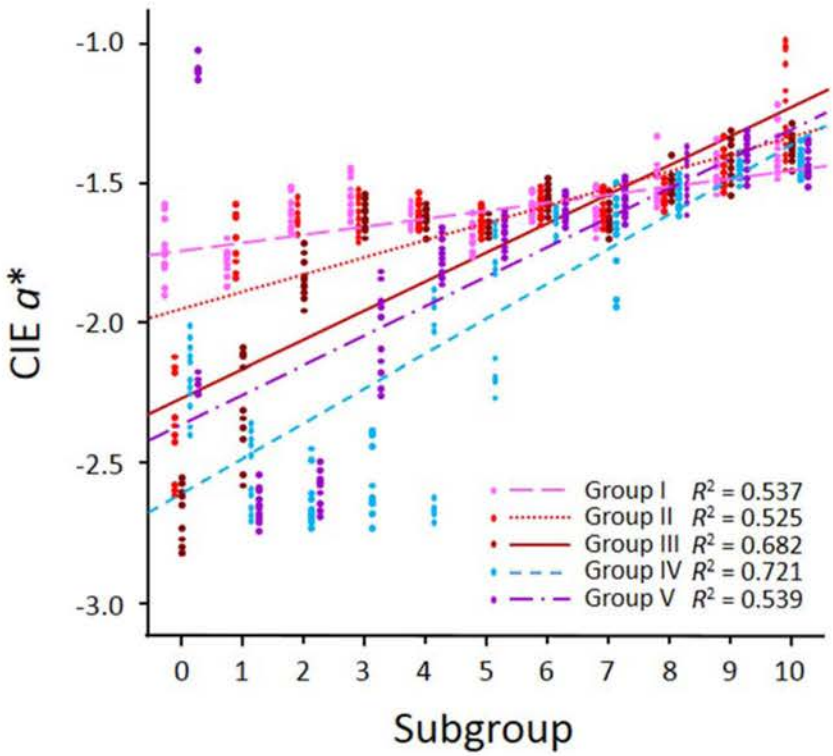

Fig. 41. Linear regression of CIE a^* values of each group as a function of the amount of thickness reduction (Experiment III).

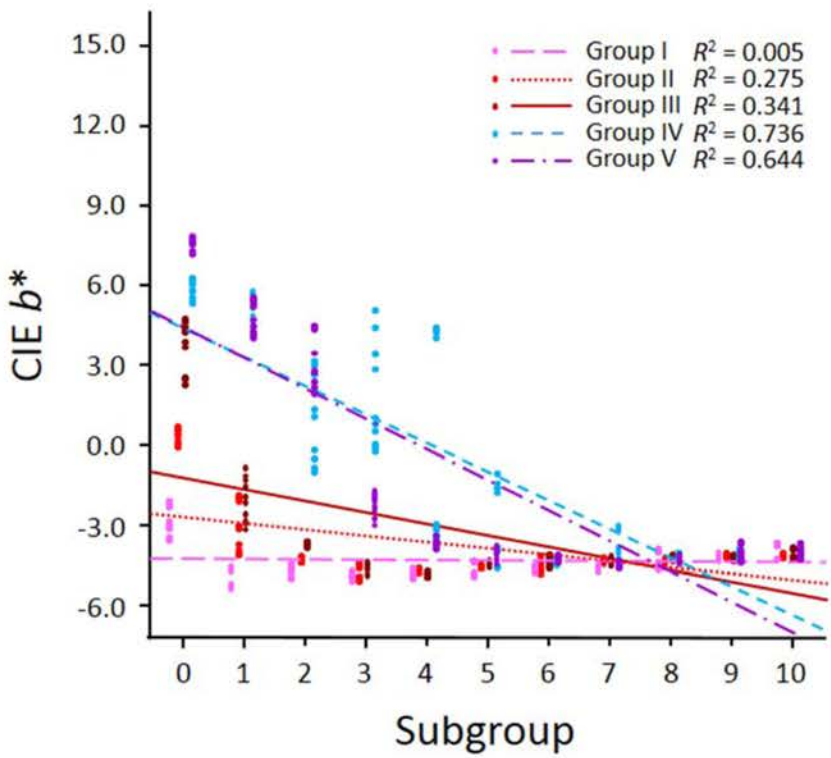

Fig. 42. Linear regression of CIE b^* values of each group as a function of the amount of thickness reduction (Experiment III).

of thickness reduction in all groups ($0.72 < r < 0.85$, $0.52 < R^2 < 0.73$, Fig. 41). There were negative correlations between b^* value and the amount of thickness reduction in all groups ($-0.86 < r < -0.07$, $0.00 < R^2 < 0.74$, Fig. 42).

Fig. 43 to 45 represented means of L^*, a^* or b^* for each group as a function of the amount of thickness reduction. Significant decrease in L^* values after initial 0.1 mm reduction was observed in all groups (Fig. 43). For a^* and b^* values, there was no significant difference between groups from *circa* 0.6 mm reduction (Fig. 44 and 45).

Average reflectance was calculated at each wavelength in the spectral range of 400 to 700 nm with an interval of 10 nm. Fig. 46 to 50 show spectral reflectance curves of average reflectance of specimens against the white background within groups. In Group I (Fig. 46), there was a significant difference between Subgroup 0 and other subgroups through the entire spectrum in the range of 400 to 700 nm and the values of spectral reflectance in other subgroups were lower than those in Subgroup 0. In Group II to IV (Fig. 47, 48 and 49), reflectance of Subgroup 0 was lower than that of other subgroups in the short wavelength range and higher in the long wavelength range. The crossing point occurred at longer wavelength with increasing the

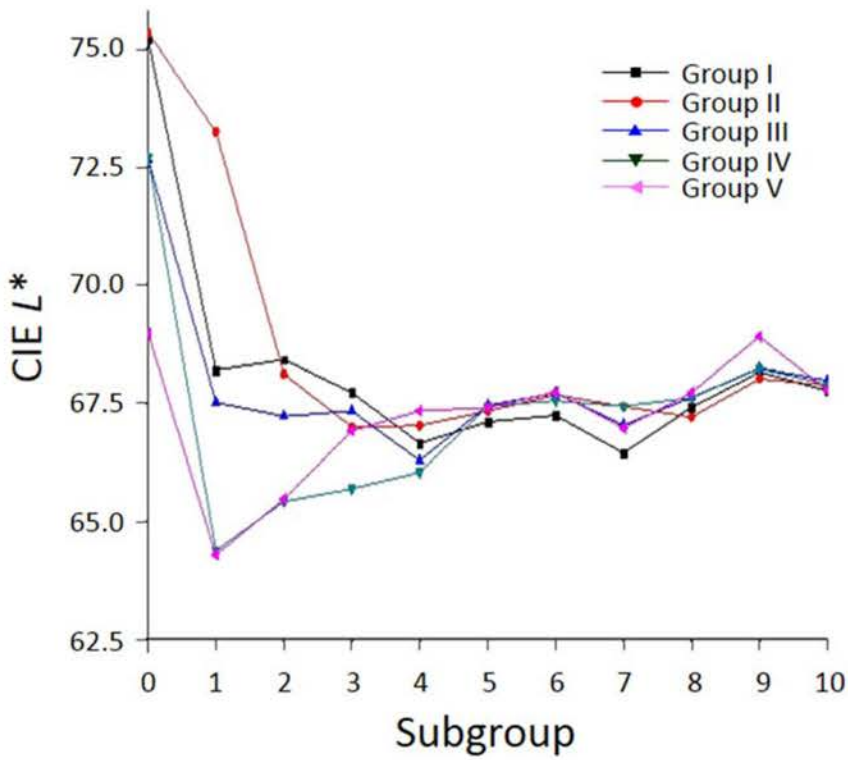

Fig. 43. Means of CIE L^* values of each group as a function of the amount of thickness reduction (Experiment III).

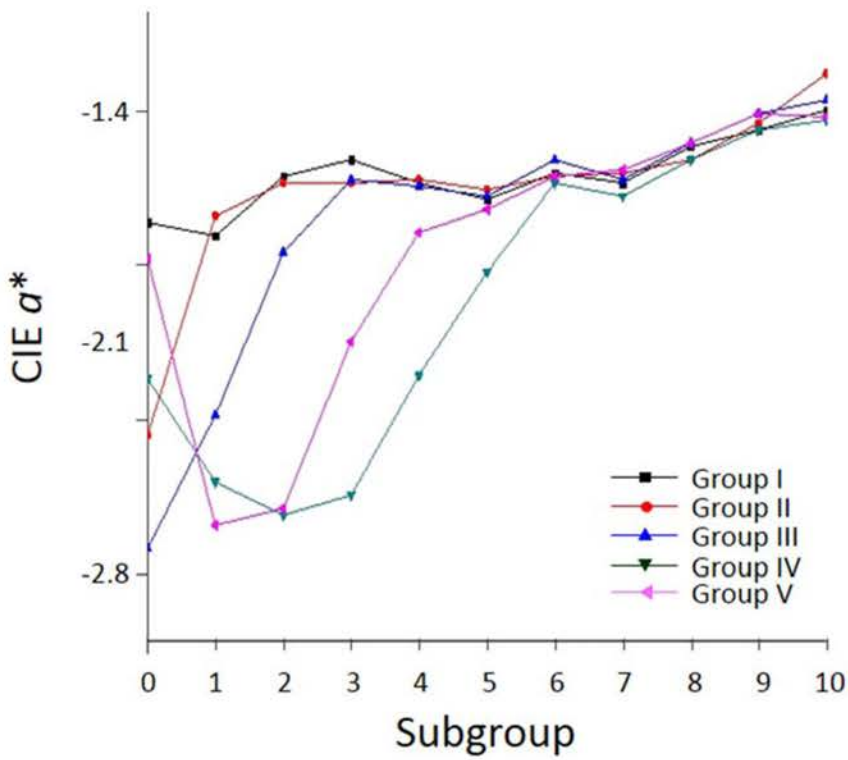

Fig. 44. Means of CIE *a** values of each group as a function of the amount of thickness reduction (Experiment III).

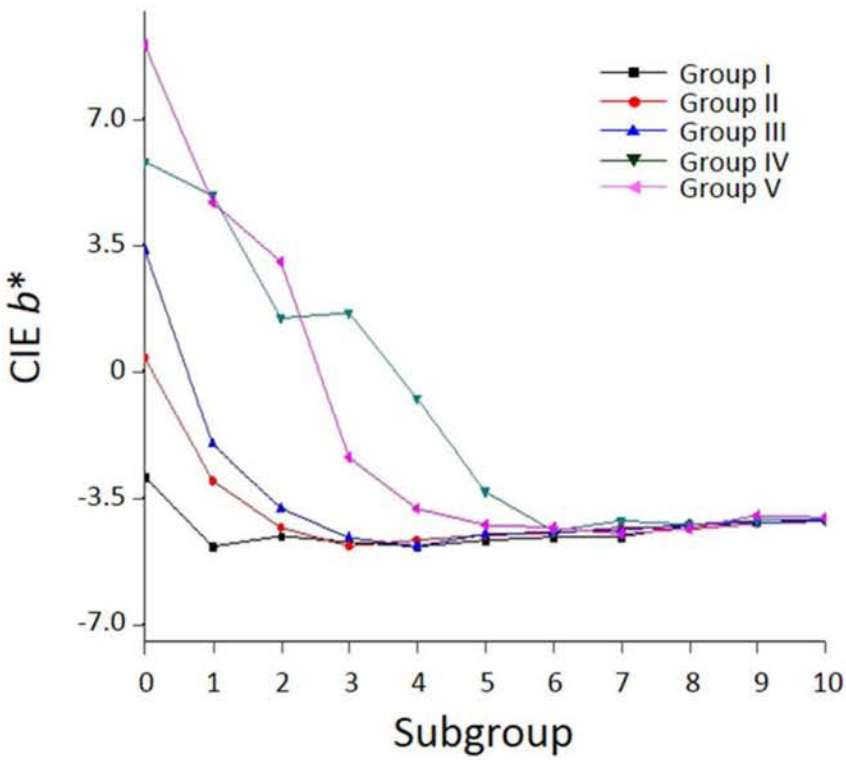

Fig. 45. Means of CIE *b** values of each group as a function of the amount of thickness reduction (Experiment III).

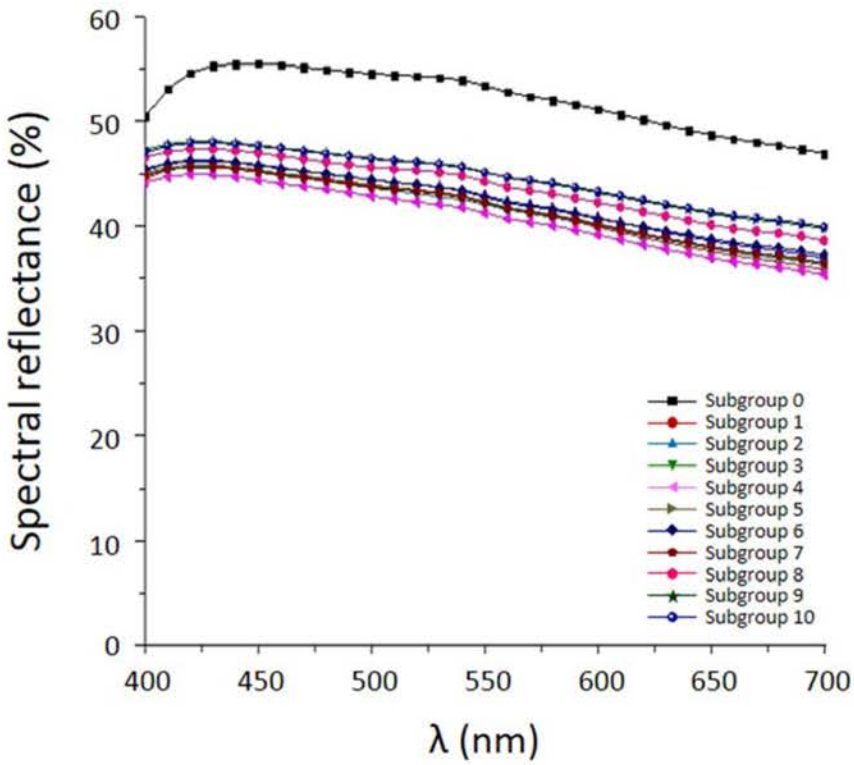

Fig. 46. Spectral reflectance of each subgroup in Group I (Experiment III).

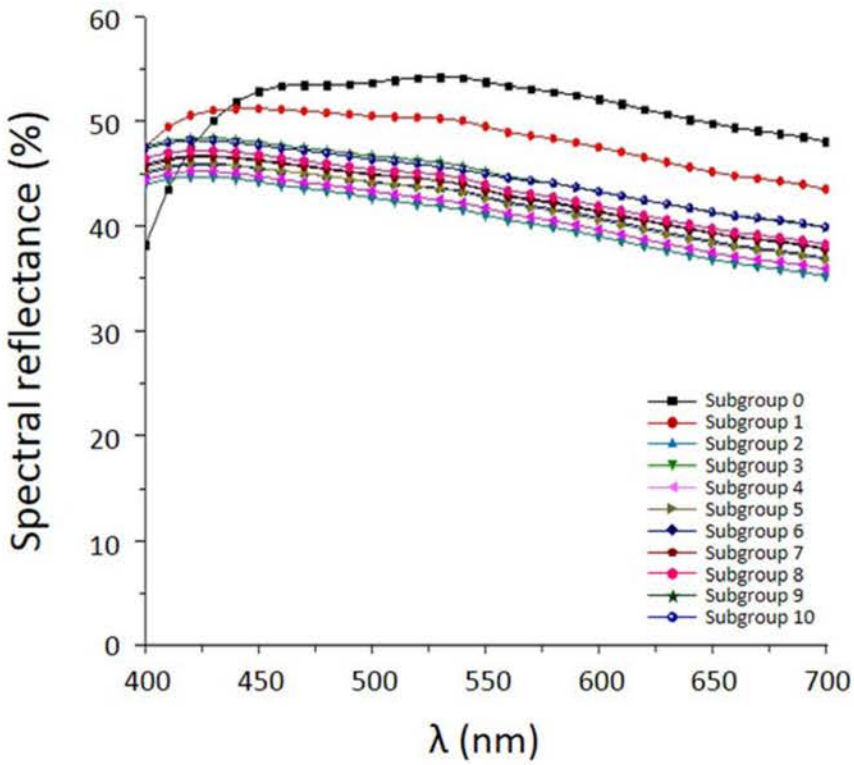

Fig. 47. Spectral reflectance of each subgroup in Group II (Experiment III).

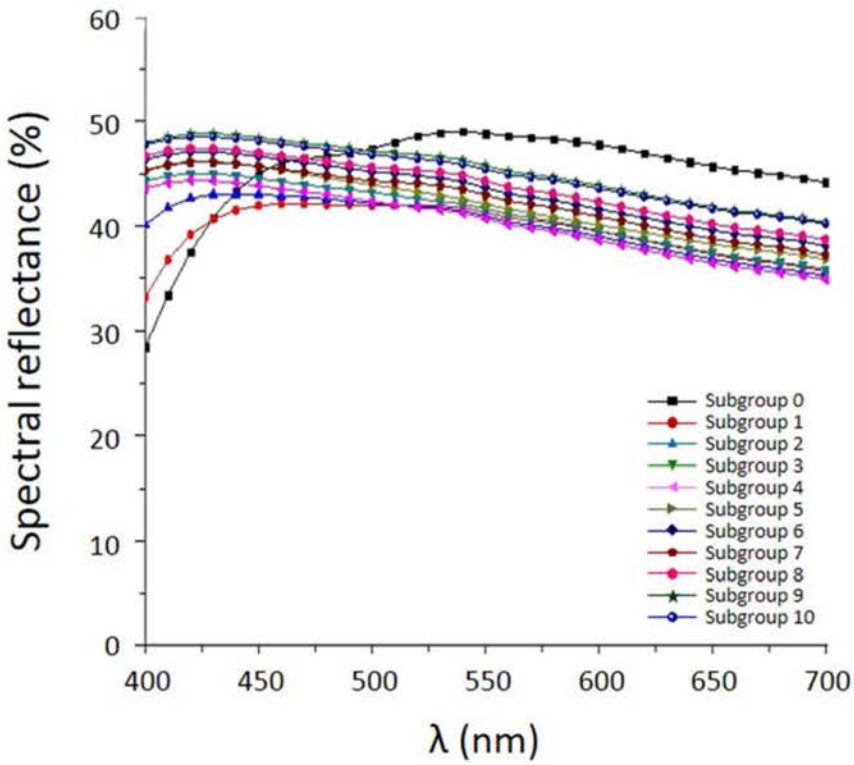

Fig. 48. Spectral reflectance of each subgroup in Group III (Experiment III).

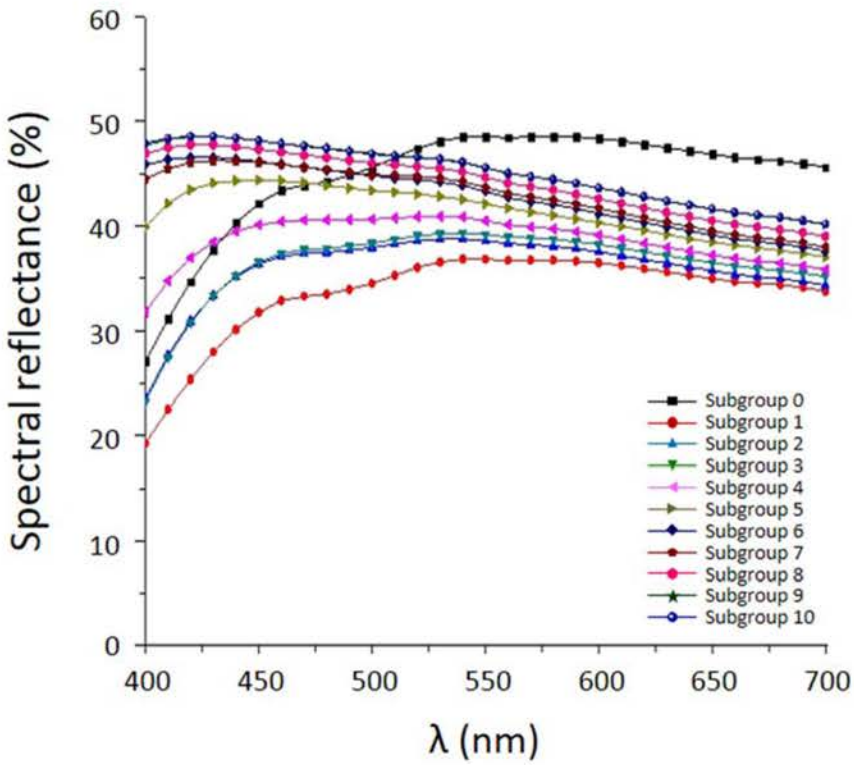

Fig. 49. Spectral reflectance of each subgroup in Group IV (Experiment III).

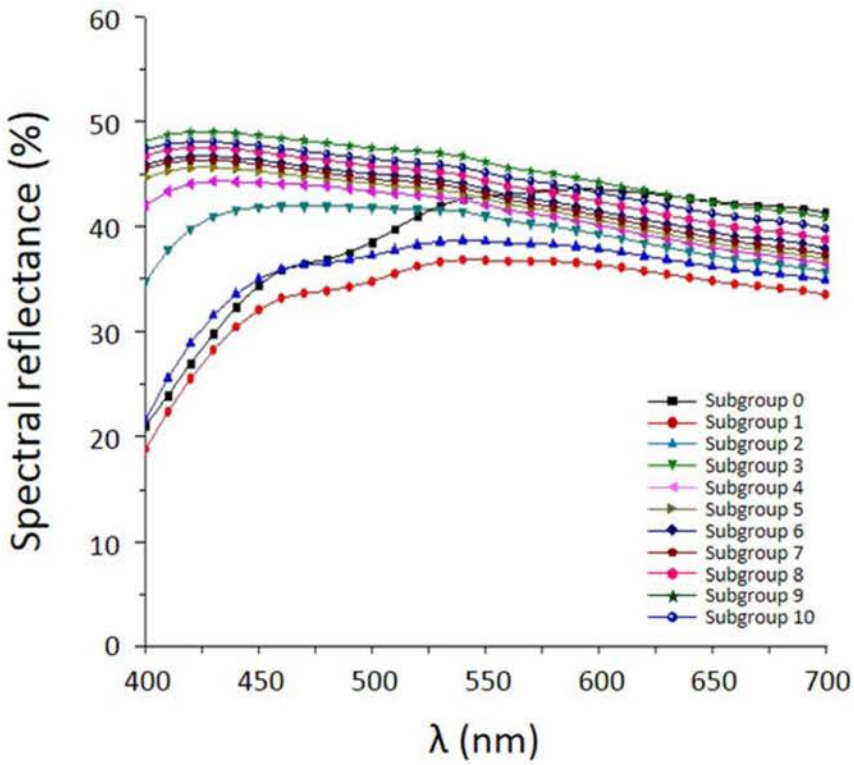

Fig. 50. Spectral reflectance of each subgroup in Group V (Experiment III).

number of coloring liquid applications. However, this trend was not so clear in Group V (Fig. 50).

Color differences (ΔE^*_{ab}) between each subgroup set are shown in Table 14. Color differences between Subgroup 0 and Subgroup 1 ranged from 4.04 to 8.38 ΔE^*_{ab} units, which were clinically perceptible ($\Delta E^*_{ab} > 3.7$). Fig. 51 shows ΔE^*_{ab} units for each group calculated by the means of L^*, a^* and b^* values between Subgroup 0 and each subgroup. Color differences between Subgroup 1 and 2 were within the range of perceptibility threshold ($\Delta E^*_{ab} < 3.7$) except Group II. Color differences between Subgroup 2 and 3 were within the range of perceptibility threshold except Group V. Color differences between Subgroup 3 and 4, 4 and 5, 5 and 6, 6 and 7, 7 and 8, 8 and 9, 9 and 10 were within the range of perceptibility threshold in all groups.

Means and standard deviations of TP for each group are listed in Table 15. TP values generally increased as the amount of thickness reduction increased in all groups (Fig. 52). Highly significant correlations were found out between TP values and the amount of thickness reduction in all groups ($r > 0.94$, $R^2 > 0.89$, $P < 0.001$, Fig. 53). Average transmittance was calculated at each wavelength in the spectral range of 400 to 700 nm with an interval of 10 nm and spectral curves are shown in Fig. 54 to 58. Each subgroup exhibited similar spectral behavior through the entire spectrum in the range of 400 to

Table 14. Color differences (ΔE^*_{ab}) between each group set

Subgroup set	Group				
	I	II	III	IV	V
0-1	7.27	4.04	7.46	8.38	6.44
0-2	6.99	8.65	9.03	8.49	6.98
0-3	7.70	9.88	9.65	8.18	11.59
0-4	8.76	9.76	10.45	9.34	12.92
0-5	8.28	9.42	9.49	10.55	13.36
0-6	8.13	9.11	9.32	11.46	13.41
0-7	8.91	9.24	9.64	11.27	13.67
0-8	7.91	9.46	9.19	11.28	13.43
0-9	7.15	8.70	8.85	10.91	13.02
0-10	7.53	8.80	8.93	11.02	13.13
1-2	0.40	5.30	1.85	3.56	2.03
1-3	0.54	6.53	2.68	3.50	7.54
1-4	1.55	6.45	3.15	5.87	9.04
1-5	1.11	6.11	2.57	8.77	9.49
1-6	1.00	5.77	2.55	9.85	9.69
1-7	1.78	5.98	2.52	9.54	9.62
1-8	1.02	6.20	2.36	9.70	9.71
1-9	0.79	5.39	2.45	9.82	9.89
1-10	0.96	5.52	2.34	9.67	9.46
2-3	0.71	1.24	0.86	0.31	5.62
2-4	1.78	1.15	1.43	2.34	7.12
2-5	1.31	0.81	0.77	5.24	7.58
2-6	1.17	0.47	0.87	6.33	7.76
2-7	1.97	0.70	0.67	6.02	7.74
2-8	1.05	0.92	0.69	6.19	7.79
2-9	0.52	0.24	1.16	6.34	7.90
2-10	0.83	0.44	0.94	6.18	7.53

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

Table 14 (Continued). Color differences (ΔE^*_{ab}) between each group set

Subgroup set	Group				
	I	II	III	IV	V
3-4	1.08	0.17	1.08	2.42	1.51
3-5	0.63	0.47	0.17	5.28	1.97
3-6	0.50	0.77	0.41	6.38	2.17
3-7	1.29	0.68	0.37	6.08	2.18
3-8	0.56	0.53	0.48	6.24	2.21
3-9	0.75	1.22	1.01	6.36	2.66
3-10	0.67	1.16	0.84	6.21	1.99
4-5	0.49	0.34	1.22	2.94	0.46
4-6	0.65	0.67	1.49	4.00	0.69
4-7	0.34	0.53	0.88	3.70	0.81
4-8	0.97	0.36	1.48	3.87	0.73
4-9	1.68	1.10	2.07	4.07	1.63
4-10	1.37	1.04	1.86	3.88	0.61
5-6	0.18	0.35	0.29	1.13	0.35
5-7	0.67	0.23	0.44	0.84	0.52
5-8	0.54	0.22	0.36	0.98	0.38
5-9	1.21	0.77	0.87	1.19	1.55
5-10	0.93	0.74	0.71	0.99	0.49
6-7	0.80	0.30	0.69	0.31	0.77
6-8	0.38	0.49	0.24	0.21	0.10
6-9	1.04	0.47	0.60	0.76	1.24
6-10	0.76	0.49	0.46	0.52	0.34
7-8	1.02	0.23	0.62	0.23	0.76
7-9	1.78	0.62	1.23	0.82	2.00
7-10	1.43	0.57	1.01	0.52	0.92
8-9	0.76	0.83	0.61	0.63	1.24
8-10	0.41	0.74	0.39	0.34	0.32
9-10	0.40	0.21	0.27	0.34	1.14

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

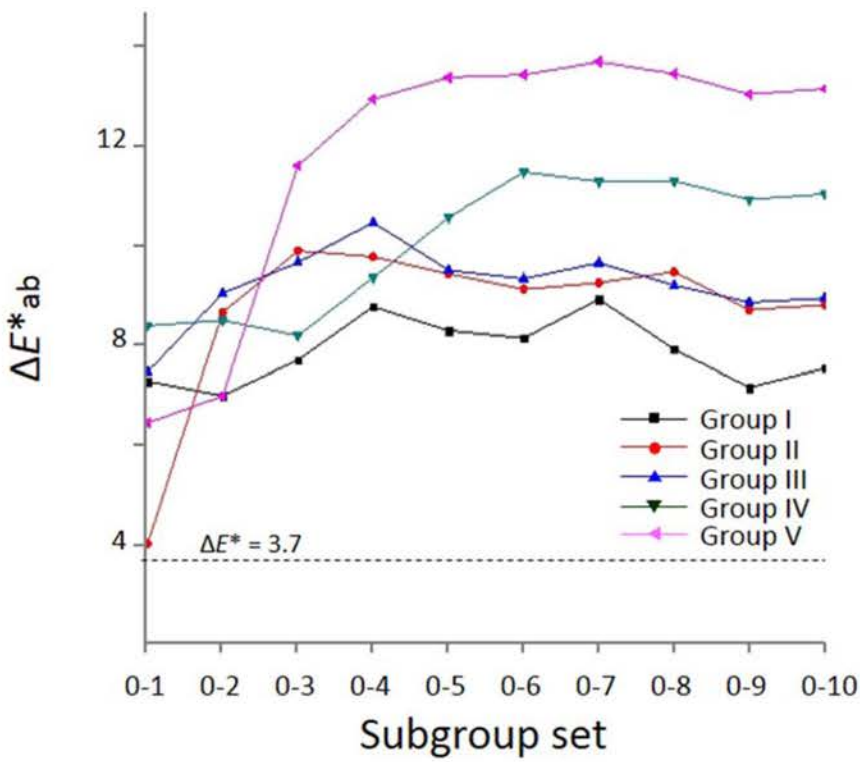

Fig. 51. ΔE^*_{ab} units between Subgroup 0 and each subgroup for each group (Experiment III).

Table 15. Means and standard deviations in parentheses for translucency parameter of each group as a function of the amount of thickness reduction

Subgroup	Group				
	I	II	III	IV	V
0	2.76[a] (0.39)	2.72[a] (0.31)	2.43 (0.10)	2.27 (0.19)	2.29 (0.13)
1	2.84[a] (0.13)	2.48[a] (0.29)	2.93[a] (0.22)	2.93[a] (0.18)	2.90[a] (0.21)
2	3.12[a,b] (0.20)	3.34[b,c] (0.09)	2.99[a] (0.13)	3.10[a,b] (0.14)	3.07[a] (0.08)
3	3.24[b] (0.06)	3.27[b] (0.12)	3.31[a,b] (0.08)	3.49[b,c] (0.22)	3.30[a,b] (0.24)
4	3.71[c] (0.10)	3.65[c,d] (0.09)	3.71[b] (0.12)	3.81[c,d] (0.17)	3.60[b,c] (0.12)
5	4.01[c,d] (0.08)	4.00[d,e] (0.13)	3.44[b] (0.35)	3.48[b,c] (0.23)	3.89[c,d] (0.11)
6	4.19[d,e] (0.09)	4.18[e,f] (0.11)	4.28[c] (0.16)	4.20[d,e] (0.08)	4.20[d] (0.09)
7	4.57[e,f] (0.18)	4.44[f] (0.25)	4.48[c,d] (0.22)	4.60[e] (0.22)	4.64[e] (0.22)
8	4.97[g,h] (0.16)	4.98[g] (0.18)	4.77[d,e] (0.27)	5.02[f] (0.27)	4.77[e] (0.32)
9	4.82[f,g] (0.18)	5.03[g] (0.33)	5.18[e,f] (0.41)	5.04[f] (0.35)	4.73[e] (0.50)
10	5.21[h] (0.58)	5.04[g] (0.41)	5.20[f] (0.48)	5.34[f] (0.52)	5.19 (0.51)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

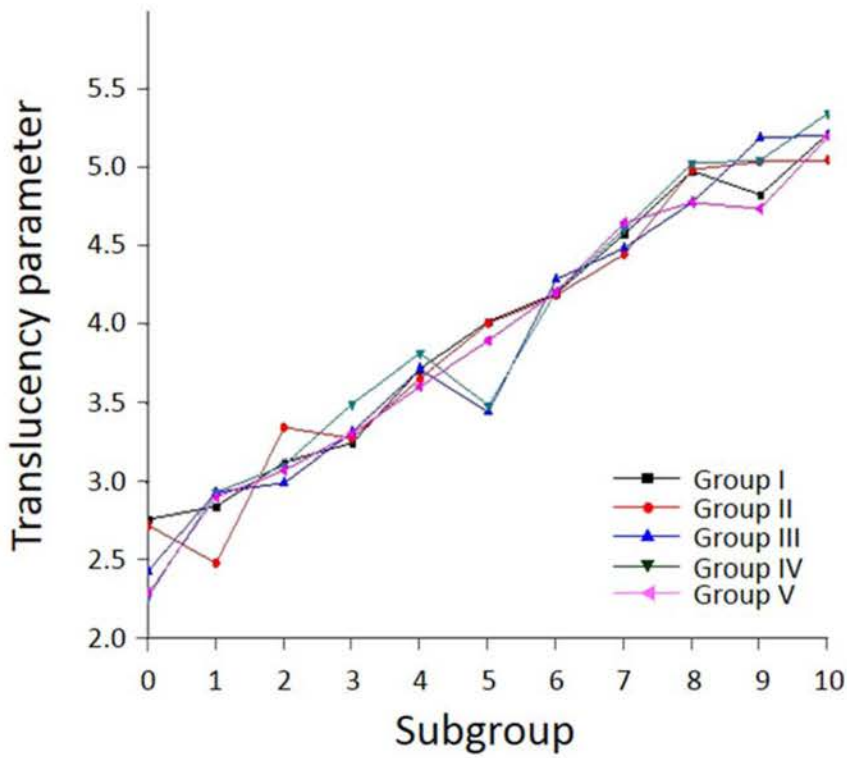

Fig. 52. Means of translucency parameter values for each group as a function of the amount of thickness reduction (Experiment III).

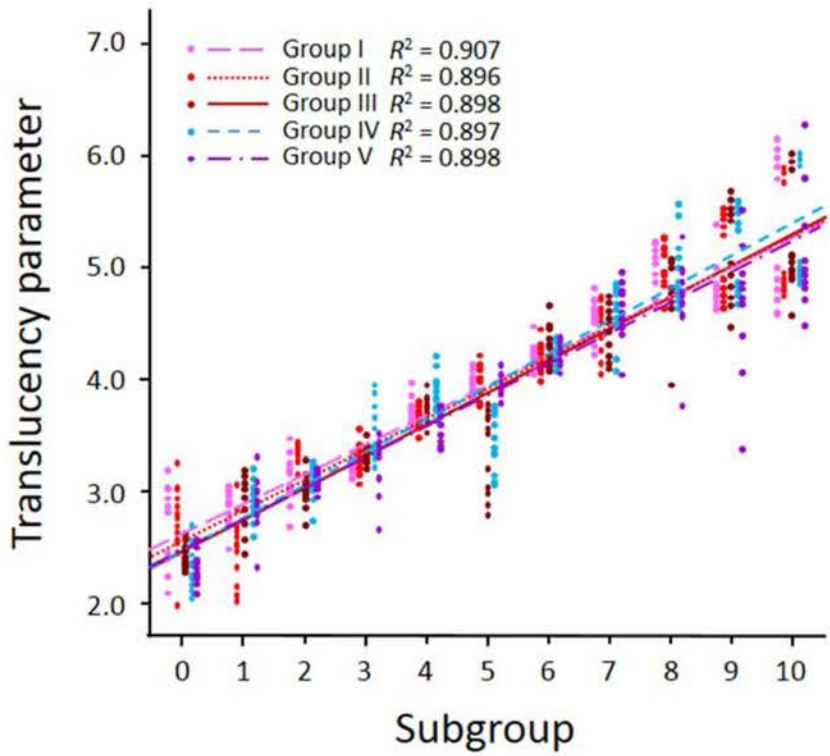

Fig. 53. Linear regression of translucency parameter values of each group as a function of the amount of thickness reduction (Experiment III).

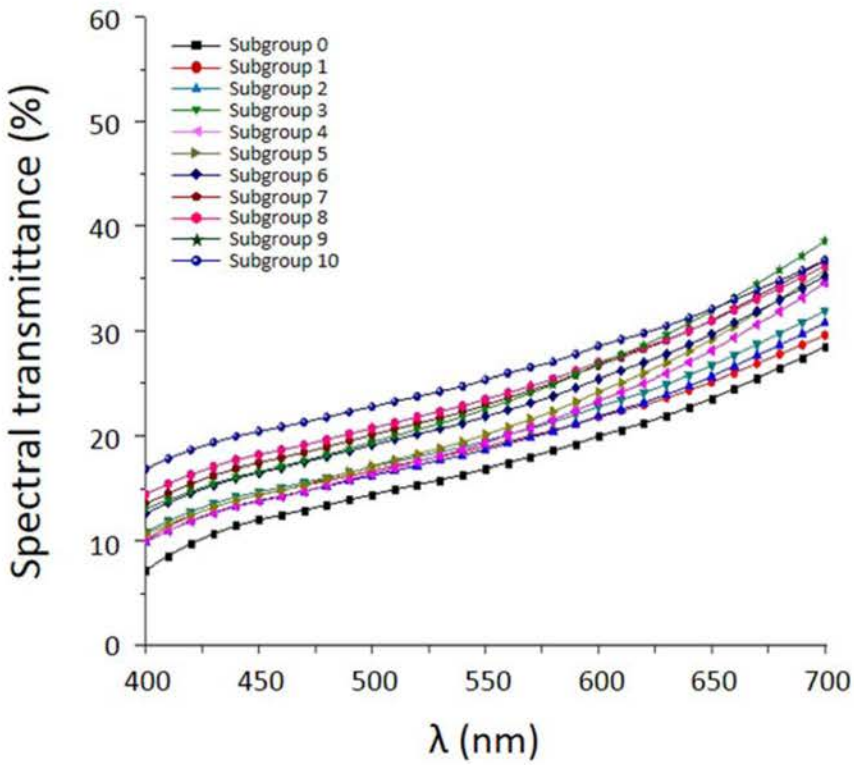

Fig. 54. Spectral reflectance of each subgroup in Group I (Experiment III).

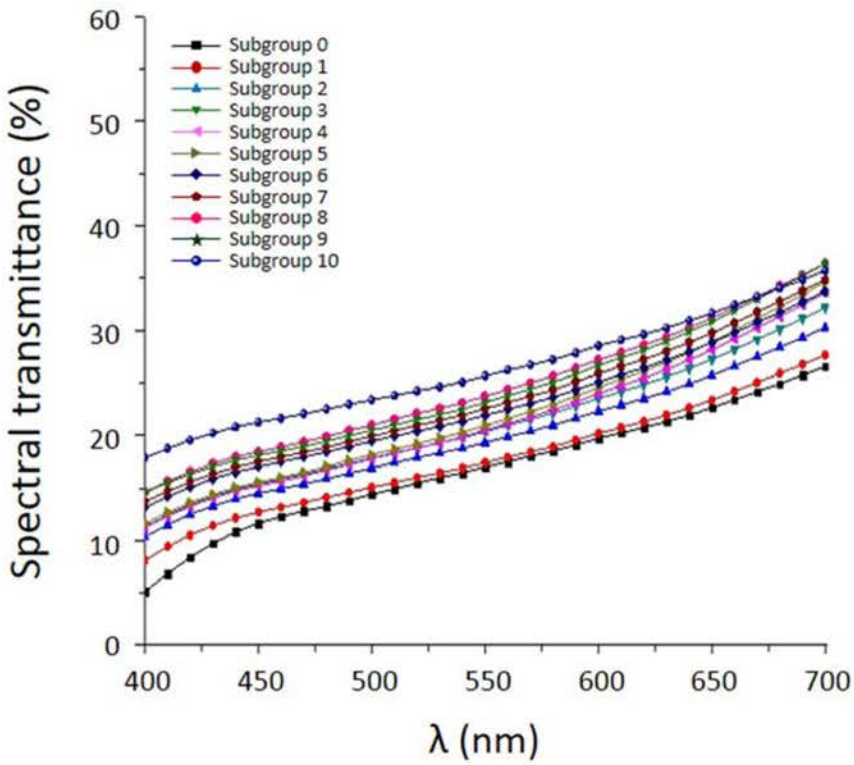

Fig. 55. Spectral reflectance of each subgroup in Group II (Experiment III).

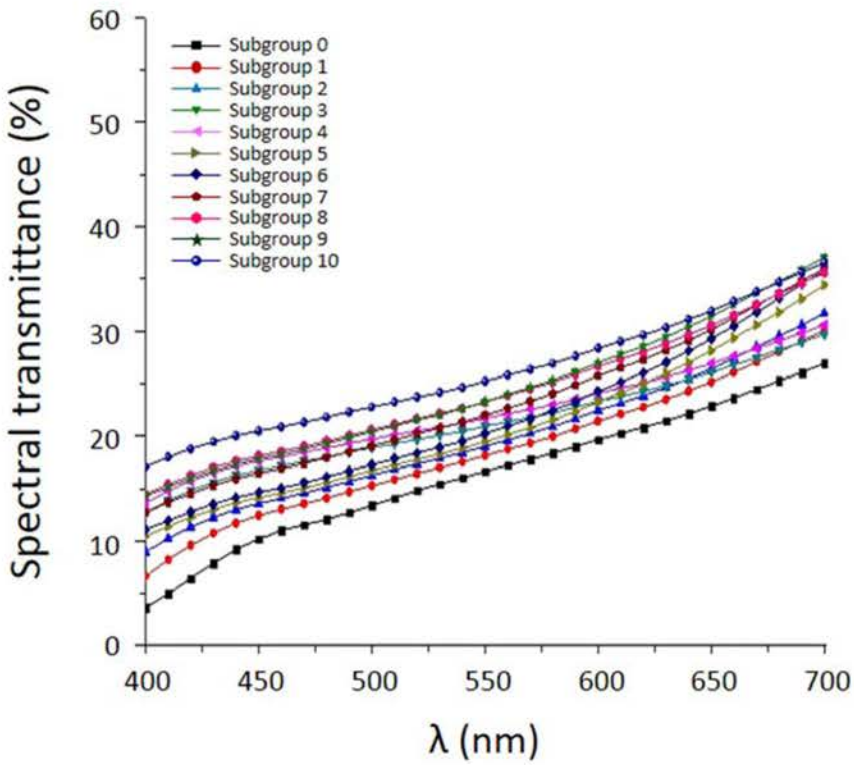

Fig. 56. Spectral reflectance of each subgroup in Group III (Experiment III).

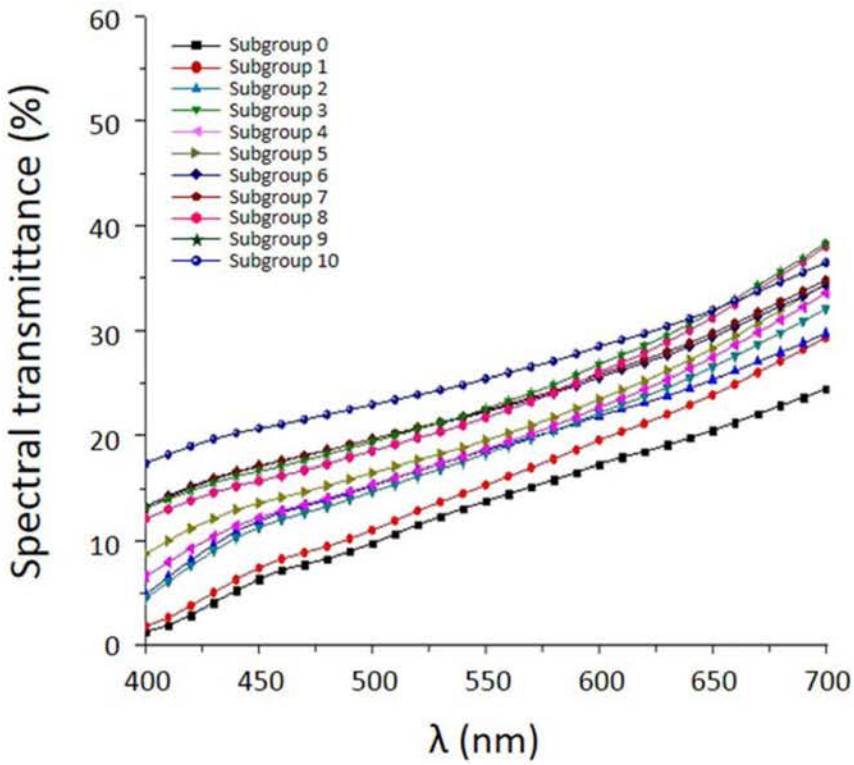

Fig. 57. Spectral reflectance of each subgroup in Group IV (Experiment III).

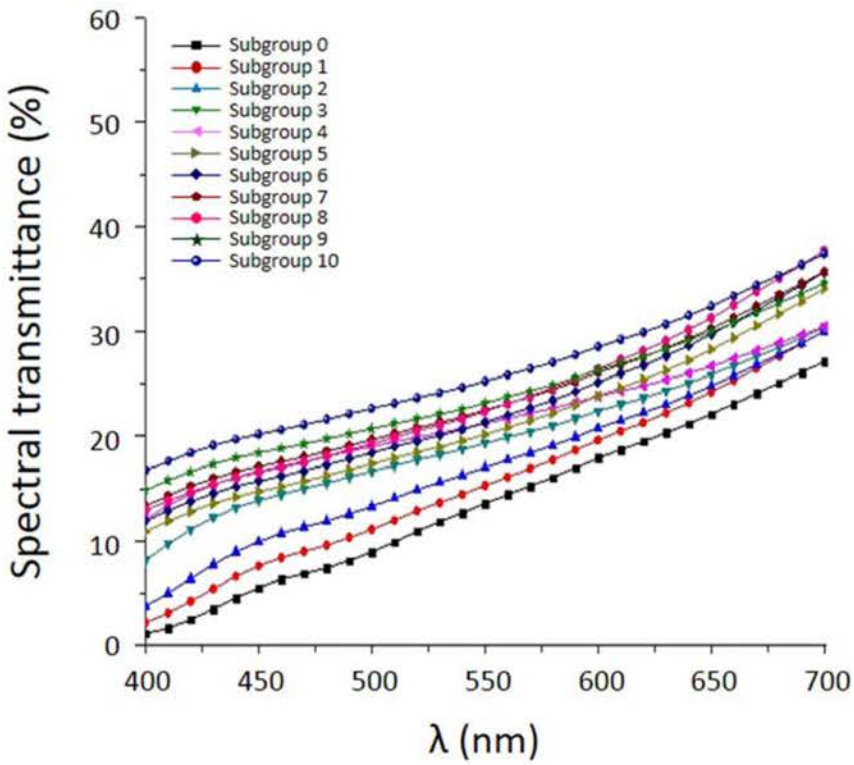

Fig. 58. Spectral reflectance of each subgroup in Group V (Experiment III).

700 nm and transmittance generally increased with increasing the amount of thickness reduction in all groups.

4. DISCUSSION

The effect of various surface modifications on the optical properties of monolithic zirconia materials was evaluated in this study. According to the result of this *in vitro* study, the null hypothesis for Experiment I, II and III could be rejected, because there were significant differences in optical properties between monolithic zirconia with different number of coloring liquid applications, different surface treatments and different amount of thickness reduction.

Based on a study by Lee *et al*. [34], CIE L^*, a^* and b^* values, relative to the standard illuminant D65, of the VITA A2 shade tab, were 52.8 ± 0.2, 0.0 ± 0.0 and 7.8 ± 0.2, respectively. In another study [35], those values of the A2 veneer-layered ceramic cores were 61.2 to 65.8, −0.5 to 1.1 and 8.8 to 12.3, respectively. According to the results of the study by Pecho *et al*. [36], those values of 0.5 mm thick human dentin were 73.3 ± 2.3, −2.1 ± 0.2 and 9.1 ± 1.2, respectively. In the present study, Table 5 shows that CIE L^*, a^* and b^* values of *circa* 2 mm thickness of control group with reflectance mode over the black background were 85.52 ± 0.33, −1.39 ± 0.94 and −1.37 ± 0.08, respectively. Thus, monolithic zirconia material is more whitish and less yellowish appearance compared to veneered ceramic [35] or natural teeth [36].

Based on the result of the present study (Table 5, 9 and 13), at least four times of coloring liquid applications might exhibit similar color values to veneered ceramic [35] or natural teeth [36]. However, direct comparison would be difficult due to different measurement protocol and material thickness.

Cho *et al*. [37] investigated the color and translucency changes of enamel porcelain after repeated staining procedures. The results showed that lightness and chroma increased, but translucency generally decreased after repeated staining. The amount of increase was dependent upon the type of stains and the number of staining cycles. Shah *et al*. [38] investigated the effect of cerium and bismuth coloring salt solutions on the color of 3Y-TZP. A perceptible color difference ($\Delta E^*_{ab} > 1$) was obtained for all test groups, where more yellowish color was identified compared to the control group. The results of the present study likewise exhibited that the increased number of coloring liquid applications with a single shade of A2 reduced lightness and increased yellowish appearance of monolithic zirconia specimen.

Color can be modified by various optical properties, such as reflection, scattering, transmission, refraction and absorption. Furthermore, surface gloss and fluorescence can also have an effect on color modifications [39]. With regard to surface texture, smooth surface could induce more light reflection [39], whereas rough surface could cause the deviation of the

reflection of specular component [40]. Obregon *et al.* [39] investigated the porcelain samples with different degree of surface roughness on the color shift. They demonstrated that different surface textures produced significant differences in hue, chroma and value. Value represented the most significant changes following the modification of surface texture with the smooth surface increasing the value. In addition, there was a shift in hue toward the yellow-red scale with the highly glazed surface. Chung [41] evaluated the effect of polishing procedures on the color and surface roughness of resin composite. In his study, polishing procedures produced a decrease in surface roughness and an increase in lightness value. Lee *et al.* [40] evaluated the effect of surface conditions on the color of dental resin composites with two different measuring geometries, i.e., SCI and SCE. They found that CIE L^* values increased after polishing with the SCE. In the study of Kim *et al.* [23], surface topography influenced especially CIE L^* value of porcelain specimens. CIE L^* value of glazed surface was lower than that of polished surface, whereas CIE a^* and b^* values increased after glazing. Color differences between polished and glazed surface were clinically perceptible ($\Delta E^*_{ab} > 3.7$). In the present study, CIE L^* values decreased after polishing and glazing. CIE L^* values showed the lowest values after polishing even though there were no statistically significant differences between polishing and glazing in some groups. For several studies with resin composites [40, 41] and feldspathic porcelains [23, 39], polishing or glazing procedures resulted in smooth

surfaces which could reflect a greater amount of light than a rough surface. As a result of reflection of incident light, lightness value increased [42]. On the other hand, in the present study, polishing or glazing decreased lightness value. Light scattering could be an important optical characteristic for translucent materials [43] (Fig. 59). Zirconia is polycrystalline structure which can induce maximum scattering effect [18] and thus, zirconia has an opaque appearance to visible light. Based on the results of the present study, surface treatments, such as polishing and glazing, seemed to reduce light scattering on the zirconia surface. Therefore, spectral reflectance decreased after polishing or glazing and lightness value decreased accordingly.

In the present study, polishing or glazing demonstrated a small shift in CIE a^* value toward green which is contrary to the previous reports [23, 39]. There was no statistical difference in CIE a^* value between polishing and glazing in Group I, II and III, while polishing showed lower CIE a^* value than glazing in Group IV and V.

In the present study, glazing increased yellowness when the number of coloring liquid applications was beyond two times. Contrary to glazing, polishing exhibited relatively stable yellow-blue color axis. Glazing procedure demonstrated more color deviation which might be related with any chemical breakdown at elevated temperature [39]. Additional firing

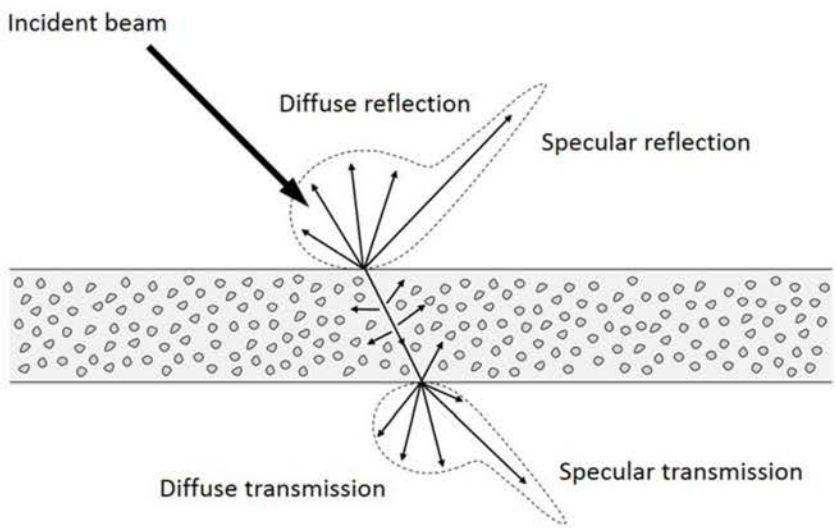

Fig. 59. Overall effect of small-particle scattering for translucent materials.

seems to cause any structural changes of monolithic zirconia. However, this needs to be evaluated in further studies. Moreover, the degree of glossiness after glazing can be controlled either by firing time or by the furnace temperature [44]. Modification of color after glazing might be different depending on the different glazing procedure.

According to the results of the present study, there were highly significant correlations between CIE b^* value and each subgroup as a function of the number of coloring liquid applications. There were negative correlations between CIE L^* value and each subgroup, whereas there were no significant correlations between CIE a^* value and Subgroup N and G. Hence, similar to the result of Experiment I, the lightness decreased and the yellowness increased as the number of coloring liquid applications increased. In addition, this tendency was not changed even after polishing or glazing procedure.

The effect of various changes in thickness of each layer on the final appearance of layered metal-ceramic structure has been studied. Jorgenson and Goodkind [45] evaluated the effect of thickness of porcelain and number of firing on color effectiveness using 1, 2 and 3 mm thickness of porcelain. In their study, no significant difference was found between hue and chroma, whereas value changes were clearly significant with the different thicknesses. As the thickness of the porcelain increased, the value increased. They

reported that the graying effect of opaque layer decreased and effect of the translucency of porcelain increased with the increase of porcelain thickness. Jacobs *et al*. [46] evaluated changes in hue, value and chroma by varying the type of metal-ceramic alloy, the dentin porcelain thickness, and the porcelain shade. They reported that there were visually and spectophtometrically differences between samples of different thickness. However, the direction of changes in value and chroma was dependent on shade and the type of metal-ceramic alloy. Terada *et al*. [25] reported color change with different thickness of dentin porcelain for metal-ceramic. They concluded that L^* value decreased and a^* and b^* value increased as the thickness of dentin porcelain increased. In the study of Lund *et al*. [47] to compare the color of textured opaque porcelains with conventional smooth surface opaque, L^* value increased and b^* value decreased with the increase of the thickness of body and incisal porcelain for both opaque porcelains in metal-ceramic systems. Douglas and Przybylska [2] investigated a dependence on thickness of dentin porcelain for shade matching using different type of dental porcelain systems and shades. They demonstrated that L^* value substantially increased with the decrease of porcelain thickness for metal ceramic systems and In-Ceram alumina system. Increasing the thickness of dentin porcelain produced more scattering and absorption of the incident light and thus, less light reflected back from the opaque layer. In their study [2], the shift in b^* value was considerably sensitive to differences in ceramic thickness, whereas the shift

in a^* value was minimal which is in accordance with the result of the present study.

In the case of metal-ceramic, light scattering and absorption within dentin porcelain layer and specular reflection at the opaque porcelain layer can affect overall color of restorations. Thus, the thickness of dentin porcelain could influence the amount of light at the opaque layer and as a result, the light reflection on the porcelain surface and back at the opaque layer could induce L^* value of the restorations.

There were several studies investigated the effect of the thickness using all-ceramic specimens. Dozic *et al.* [28] determined the effect of different thickness ratio of opaque and translucent porcelain on the overall color of all-ceramic specimens for A1, A2 and A3 shade. Both a^* and b^* values increased as the thickness of opaque layer increased for all shades. L^* value was shade dependent. Shokry *et al.* [27] investigated the effect of the different thickness ratio of core and veneer of two all-ceramic systems, such as IPS Empress and In-Ceram Spinell, on the color parameters of layered specimens. In their study, L^* value decreased while a^* and b^* values increased as the total specimen thickness increased for both ceramic systems. In addition, core and veneer interaction as well as the thickness strongly influenced overall color of layered ceramic specimens. They explained that increased absorption of

incident light with thicker specimens reflected reduced quantity of light and accordingly, L^* value decreased. Furthermore, they demonstrated that translucency of dental ceramic was often related with lower L^* value. Ozturk *et al*. [29] evaluated the effects of various dentin ceramic thicknesses (0.5, 1 or 1.5 mm) with a core thickness of 1 mm and number of firings on the color of lithium disilicate glass-ceramic and zirconium oxide all-ceramic systems. In their study, L^* value significantly decreased as the veneering ceramic thickness increased for IPS e.max Press and DC-Zirkon specimens. There were significant increases in a^* and b^* values with the increase of the thickness for IPS e.max Press specimens. For DC-Zirkon specimens, there was a significant increase in a^* value, while no significant difference in b^* value as the ceramic thickness increased. The present study has shown that there was a significant decrease in L^* value after initial 0.1 mm reduction, but there were no distinct changes from 0.2 mm to 1.0 mm reduction in most groups.

With regard to all-ceramic restorations, the amount of light reflection at the opaque core seems to be different between all-ceramic systems with different core translucency although L^* value generally decreased with the increased thickness of veneering porcelain due to the increased absorption of incident light. Douglas and Przybylska [2] demonstrated that due to the lack of opaque

core, the semi-translucent all-ceramic porcelain systems were less affected by reduced porcelain thickness compared to metal-ceramic for shade matching.

Incident light that is not reflected at the surface layer suffer internal reflection by reversing the direction and may be seen by the inspector [48] (Fig. 60). In the present study, the changes in L^* value showed different aspect compared to metal-ceramic and all-ceramic systems. The possible reason would be the difference of layering structure, since monolithic zirconia consists of a single opaque layer. Based on the results of the present study, it can be inferred that there might be reduced scattering due to the reduced thickness which induced lower L^* value at first 0.1 mm reduction. However, as the thickness reduction proceed, monolithic zirconia itself acts as an opaque core and could induce internal reflection. With the increase of the thickness reduction, reduced reflection might compensate for increased internal reflection and thus, L^* value could be relatively stable up to 1.0 mm reduction ($-0.59 < r < 0.35$, $R^2 < 0.35$, Fig. 40). Colorant might affect scattering and absorption *circa* 0.4 mm deep in the specimens and there seems to be no additional colorant effect on L^* value (Fig. 43). Consequently, clinicians can predict that there might be a small decrease in L^* value after initial reduction in a clinical situation, but L^* value could remain stable until 1.0 mm of thickness reduction. In addition, further study should be required to determine whether there is any difference in L^* value when the thickness reduction is more than 1.0 mm.

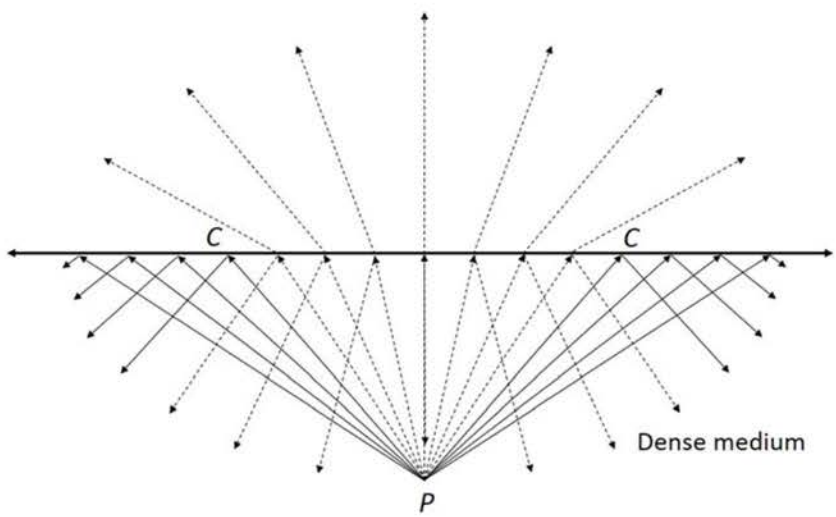

Fig. 60. When a light passes from a material with a higher refractive index to a lower refractive index, total internal reflection can occur. *P*: pigment particle. *C*: critical angle.

Chromatically, a^* value substantially increased, while b^* value generally decreased with the decrease of the thickness. In terms of the shift in chroma, b^* value was more sensitive to the change of thickness than a^* value showing minimal shifts in a^* value. This result is in accordance with previous studies [2, 28, 29]. According to the study of Douglas and Brewer [49], dental observers were more sensitive and critical to the color difference in redness than yellowness for metal-ceramic crowns. Therefore, perception of color difference in yellowness might be somewhat tolerant toward thickness changes compared to redness for human observer. However, the previous study [49] was conducted with metal-ceramic crowns and therefore, further study should be performed to determine whether there is any difference in subjective color assessment between a^* and b^* value for monolithic zirconia restorations.

As a result, it can be inferred that the color of ceramic restoration is influenced by its thickness regardless of the ceramic systems and the direction of the changes of L^*, a^* or b^* value was dependent on the type of the material tested. In the present study (Fig. 44 and 45), Group I joined with other graphs at 0.1 mm reduction, and Group II joined at 0.2 mm reduction, and Group III joined at 0.3 mm reduction, and Group IV joined at 0.6 mm reduction, and Group V joined at 0.6 mm reduction for both a^* and b^* value. Based on the results of the present study, it can be inferred that one time of application of coloring

liquid could infiltrate 0.1 mm deep through monolithic zirconia, two times of application could infiltrate 0.2 mm deep, three times of application could infiltrate 0.3 mm deep, four times of application could infiltrate 0.6 mm deep, five times of application could infiltrate 0.6 mm deep through monolithic zirconia.

Several studies [50-54] have attempted to investigate the threshold for perceptibility and acceptability of color difference. Johnston and Kao [50] determined 3.7 ΔE^* units as a perceptibility threshold and 6.8 ΔE^* units as a borderline for color match or mismatch between composite veneers and teeth. Other *in vivo* study [51] indicated 2.6 ΔE^* units as 50/50 perceptibility of color difference. This perceptibility threshold was different from those of other *in vitro* studies which identified 1 ΔE^* unit [52] and 2 ΔE^* units [53]. Ghinea *et al*. [54] reported that a 50% perceptibility threshold was 1.8 ΔE^* units using dental ceramic discs. The interpretation of the color difference for the present study is based on the visual matching study of Johnston and Kao [50].

In the present study, color differences for all colored groups when compared to the control group in Experiment I were above a clinically perceptible level ($\Delta E^*_{ab} > 3.7$), while there were no perceptible color changes between two subsequent groups.

Color differences between no treatment and polishing was higher than between no treatment and glazing in Experiment II of the present study. This would be caused by the higher difference of lightness value between no treatment and polishing. This is in accordance with Chung's study [41] which demonstrated that color difference was mainly determined by the lightness rather than the hue and chroma. Color differences between no treatment and polishing can be perceived in a clinical setting ($\Delta E^*_{ab} > 3.7$). Color difference between no treatment and glazing can also be detectable in a clinical setting. Thus, surface treatment, whether polishing or glazing, could modify the color interpretation. However, there were no perceptible color differences between polishing and glazing in most groups, which means that human eye cannot detect the color difference between these two procedures.

Based on the results of Experiment III in the present study, color differences can be perceived in a clinical setting ($\Delta E^*_{ab} > 3.7$) even after first 0.1 mm reduction regardless of the number of coloring liquid applications. In addition, when the thickness was reduced by 0.3 mm or more, larger color difference compared with no reduction was perceived as the number of coloring liquid applications increased (Fig. 51).

Consequently, various surface modifications, such as coloring procedure, polishing or glazing, and thickness reduction could induce noticeable shift in

color parameters. Since high value of monolithic zirconia yield a less vital appearance, reduced value resulted from coloring, polishing and glazing might improve natural-looking appearance. Glazing could improve yellowish appearance of monolithic zirconia. However, loss of glaze layer could occur within the first six months after insertion of the restorations [55]. Since there was no perceptible color difference between polishing and glazing, polishing could be recommended which seems to be more stable in terms of long-term color appearance. Thickness reduction could reduce brightness and produce reddish and bluish appearance of monolithic zirconia. Therefore, this tendency should be taken into account during the adjustment procedure by the dentist to achieve an optimal occlusal contact. In addition, thickness reduction could induce wider range of the shift in b^* values than in a^* values and thus, this should be considered in color matching as well.

In the present study, the statistical analyses showed that the influence of the number of coloring liquid applications, polishing and glazing on TP value was not significant. There was a study [56] which showed that there were significant differences in contrast ratios of Procera zirconia between specific shades. Pecho *et al*. [36] evaluated the translucency of both non-colored and colored zirconia, and compared them with human and bovine dentin of 0.5 mm thickness. They exhibited that TP values of human dentin, bovine dentin and zirconia showed no significant differences among them, indicating

17.2 ± 1.8 for human dentin and 17.0 ± 1.7 for bovine dentin. No significant differences were also found between non-colored and colored zirconia systems. Yu *et al*. [57] investigated the translucency of human and bovine enamel and dentin. Mean TP values of 1 mm thick bovine enamel, bovine dentin, human enamel and human dentin were 14.7, 15.2, 18.7 and 16.4, respectively. In the present study, TP values of monolithic zirconia specimens of 2 mm in thickness ranged from 9.15 to 11.69 in Experiment I. Experiment II and III exhibited lower TP values than Experiment I and this might be due to the edge loss effect of the spectrophotometer. The configuration of spectrophotometer is important in the measurement of color and translucency. Bolt *et al*. [58] and ten Bosch and Coops [59] described that the re-emitted photon was scattered beyond the edge of the opening especially for translucent material, and the smaller window area view caused the greater amount of edge-loss. In an attempt to reduce the edge-loss, the window size of 19 mm in diameter and the reduced beam size of 1 mm × 5 mm were used in Experiment I of the present study.

Jiang *et al.* [60] reported that sintering temperature and particle size significantly affected the density and transmittance of Y-TZP and the smaller nanoparticles had higher transmittance than that of larger particles. In addition, the presence of impurities and the sintering conditions, such as sintering temperature and time, could affect significantly on the grain size [61, 62].

There have been several sintering methods which have tried to derive nanocrystalline ceramic with a full density, such as spark-plasma-sintering [63], hot pressing [64], and two-step sintering procedure [65], etc. Casolco *et al*. [66] had attempted to obtain translucent zirconia ceramics combining nanopowders with nanocrystalline grain sizes of full density. They suggested that when the grain size is significantly smaller than the wavelength of visible light, there would be more transmission of light rather than a scattering caused by interaction with internal particles.

Based on the results of the present study, translucency of monolithic zirconia is dependent on the thickness, and light transmission is a function of its thickness as well. The present study measured total transmission including scattering using the spectrophotometer with an integrating sphere [67]. Fig. 32-39 and Fig. 54-58 showed that transmittance generally increased with the increase of incident wavelength. As stated by the Rayleigh scattering equation [68], scattering of incident light seems to decrease in monolithic zirconia with increasing wavelength.

The effect of the thickness on the translucency of ceramic restorations was studied in all-ceramic systems. Antonson and Anusavice [30] investigated the contrast ratio of dental core and veneering ceramics as a function of ceramic thickness. There was a relatively strong positive linear correlation between

CR and thickness ($R^2 > 0.81$). Hefffernan *et al*. investigated the effect of the thickness of a core material [43] on its translucency, and the thickness of a veneered core material [69] on the overall translucency of the specimens. They demonstrated that increased thickness resulted in greater opacity. O'Keefe *et al.* [70] suggested that that the thickness of the porcelain veneer was the primary factor affecting light transmission and not the opacity. The present study demonstrated similar results. TP values generally increased as the amount of thickness reduction increased in all groups ($r > 0.94$, $R^2 > 0.89$, $P < 0.001$, Fig. 53). Based on Lambert's law which states that equal amounts of absorption occur in equal thicknesses of material [68], decreasing the thickness of material allow greater amount of light transmission. Furthermore, the fraction of incident light that are reflected, absorbed, and transmitted depend on the thickness of the specimen as well as the scattering and absorption characteristics [71]. Accordingly, translucency of monolithic zirconia restoration could be controlled by its thickness as well as the application of efficient sintering procedures and grain size [60, 63-66] rather than coloring procedure, polishing or glazing.

Opalescence can improve the natural appearance and the vitality of restorations. For opalescence feature, there should be a light scattering of shorter wavelengths of the visible spectrum in a translucent material. Opalescent material appears blue in reflected light and orange-yellow in

transmitted light [72]. Based on the previous study [73], OP value of the commercial resin composite specimens of 1 mm in thickness was in the range from 5.7 to 23.7, which was varied by their brand and shades. Another study [74] reported that OP values of bovine enamel of 0.7 mm-1.1 mm in thickness ranged from 7.6 to 22.7, which was varied by the configuration of spectrophotometers and those of human enamel of 0.9 mm-1.3 mm in thickness ranged from 19.8 to 27.6. Cho *et al.* [75] investigated the opalescence of all-ceramic core and veneer materials using a spectrophotometer. In their study, the range of OP value was 1.6-6.1, 2.0-7.1, 1.3-5.0 and 1.6-4.2 for the core, veneer, A2- and A3-layered specimens, respectively, and all of which were significantly influenced by the type of materials. According to one of the US patents [33], the OP value was represented based on 1 mm thick resin composite specimens as a difference in the chromaticity [$\Delta C^*_{ab} = (\Delta a^{*2} + \Delta b^{*2})^{1/2}$] between the reflected and transmitted colors. It was reported that the OP value should be at least *circa* 9 to contribute the vitality of dental restorative composites. The restorative materials with OP values of between 4 and 9 may be considered to have some opalescence only slightly discernible to the naked eyes. Based on the results of the present study, when the number of coloring liquid applications is more than three times, monolithic zirconia restorations could be regarded non-opalescent under this criterion.

According to the results of the present study, there was a highly significant correlation between OP and Δb^* ($r = -0.782$). This means that OP was mainly influenced by the change in CIE b^*. This is in accordance with the several studies [74, 76] on opalescence, which demonstrated that OP value was correlated with Δb^* and ΔE^*_{ab}. However, there is a study [75] which showed that the correlations between OP and ΔE^*_{ab}, or between OP and Δb^* were not significant for layered all ceramic specimens. On the other hand, another study [73] exhibited that there was a weak correlation between OP and Δa^* ($R^2 = 0.076$). Several studies [73, 77] demonstrated that there was a strong correlation between TP and OP. However, no correlation was found in the present study. Since the presence of small internal particles influences maximizing opalescence in a translucent material [78], grain size might have a significant effect on the translucency and opalescence of monolithic zirconia restorations.

Esthetic demand is increasing for monolithic zirconia restorations. Fabricating monolithic zirconia restorations with an appropriate shade, translucency and opalescence is becoming a major concern for a natural-looking appearance. Surface modifications to improve esthetic outcome, such as coloring procedure, polishing and glazing, and thickness reduction followed by adjustment procedure could be possible factors that affect overall appearance of monolithic zirconia restorations. Considering the prediction

and reproduction of overall appearance of monolithic zirconia restorations, clinicians should take into account the possible color deviations after coloring procedure, polishing or glazing, and thickness reduction. Furthermore, different shade guides considering any color changes following surface modifications can be helpful for monolithic zirconia restorations.

This *in vitro* study has several potential limitations. The first limitation is that even application of coloring liquid on the specimens, uniform application of glazing paste without any void, and exact amount of the increase of coloring liquid applications were difficult to control. Secondly, possible pressure fluctuation inside the furnace during sintering process might induce uneven color of the specimens. In addition, possible temperature fluctuation inside the furnace might not yield homogenous glazing. Thirdly, the aperture diameter of spectrophotometer used for Experiment II and III in the present study for reflectance measurement was 3 mm and possible edge loss would affect color measurement. Finally, this study was conducted with limited color of shade A2 and with a specific kind of monolithic zirconia system and coloring liquid. Therefore, the influence of varied color combinations with different monolithic zirconia systems should be further studied. In addition, further study is needed to investigate the effect of additional surface treatment after thickness reduction by the adjustment procedure on the color and translucency of monolithic zirconia restorations. Moreover, further study

should consider *in-vivo* overall color shift which might be related with the color of underlying cement and tooth substrate.

For the clinical application of monolithic zirconia, based on the results of the present study, at least four times application of coloring liquid could be recommended considering the change of the optical properties according to the surface modifications. Especially, for the application in anterior region, additional blue coloring could be considered since monolithic zirconia has little opalescence property. In addition, polishing would be recommended rather than glazing for monolithic zirconia restorations concerning long-term color stability. Furthermore, the thickness of monolithic zirconia restorations could be determined considering the translucency especially for the discoloration of the tooth substructure.

5. CONCLUSION

Within the limitations of this study, the following conclusions can be drawn. Increasing the number of coloring liquid applications results in a decrease of the lightness and an increase of the yellowish appearance of monolithic zirconia. Polishing decreases the lightness and glazing also decreases the lightness, while it increases the yellowish appearance of monolithic zirconia. Increasing thickness reduction decreases lightness and increases the reddish appearance and decrease the yellowish appearance of monolithic zirconia. In addition, reduced thickness produces more translucent monolithic zirconia. Clinicians can predict that there would be a shift in optical properties of monolithic zirconia restorations with various surface modifications, such as coloring, polishing, glazing and thickness reduction.

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국문 초록

다양한 표면 변화가 치과용 단일 구조 지르코니아 수복물의 광학적 특성에 미치는 효과에 관한 연구

서울대학교 대학원 치의과학과 치과보철학 전공

(지도교수 이 재 봉)

김 희 경

연구 목적: 다양한 표면 변화가 단일 구조 지르코니아의 광학적 특성에 미치는 효과에 관하여 알아보고자 하였다; 1) 염색 횟수가 단일 구조 지르코니아의 광학적 특성에 미치는 효과, 2) 연마와 광택 처리가 단일 구조 지르코니아의 색상과 투명도에 미치는 효과, 3) 다양한 삭제량이 단일 구조 지르코니아의 색상과 투명도에 미치는 효과.

재료 및 방법: 단일 구조 지르코니아로 BruxZir, 염색액으로 A2 색상의 Tanaka ZirColor를 선택하였다. 첫 번째 실험에서는 총 18개의 지르코니아 시편(가로 27.6mm, 세로 27.6mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 6개의 그룹(그룹 O 에서 그룹 V)으로 나누고 각각 색상, 색 차이, 투명도 및 유백광도를 측정하였다. 두 번째 실험에서는 총 45개의 지르코니아 시편(가로 16.3mm, 세로 16.4 mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 5개의 그룹(그룹 I 에서 그룹 V)으로 나누고, 각 그룹을 표면 처리 방법에 따라 3개의 소그룹(대조군(N), 연마군(P), 광택군(G))으로 나눈 후, 각 시편의 색상, 색 차이 및 투명도를 측정하였다. 세 번째 실험에서는 총 165개의 지르코니아 시편(가로 16.3mm, 세로 16.3mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 5개의 그룹(그룹 I 에서 그룹 V)으로 나누고, 각 그룹을 두께 삭제량에 따라 0.1mm 간격으로 1.0mm 삭제까지 11개의 소그룹(소그룹 0 에서 소그룹 10)으로 나눈 후, 각 시편의 색상, 색 차이 및 투명도를 측

정하였다. 모든 측정은 CIELAB 색공간을 사용하였고, CIE 표준 광원 D65에서 분광광도계를 사용하여 한 시편 당 다섯 부위를 측정하였다. 통계는 SPSS 프로그램을 이용하여 일원분산분석 및 Scheffé 다중검정을 실시하였으며, 각각의 측정값 간의 유의성 및 상관관계도 알아보았다 (a = 0.05).

결 과: 첫 번째 실험에서는 염색 횟수가 증가함에 따라 CIE L^* 값과 유백광도는 감소하였으며, CIE b^* 값은 증가하였다. CIELAB 색차는 1.3 에서 15.7 사이로 나타났으며, 투명도의 유의한 변화는 없었다. 두 번째 실험에서는 모든 그룹에서 N과 P간에 CIE L^* 값의 유의한 차이를 보였으며, P와 G간에 CIE b^* 값의 유의한 차이를 보였다. 모든 그룹에서 N과 P간의 색 차이를 지각할 수 있었으며, 대부분의 그룹에서 N과 G간의 색 차이를 지각할 수 있었다($\Delta E^*_{ab} > 3.7$). 대부분의 그룹에서 각 소그룹 간의 투명도의 차이는 나타나지 않았다. 세 번째 실험에서는 삭제량의 증가에 따라 CIE L^* 과 CIE b^* 값이 감소하였으며, CIE a^* 값은 증가하였다. 모든 그룹에서 소그룹 0과 1간의 색 차이를 지각할 수 있었으며, 모든 그룹에서 삭제량의 증가에 따라 투명도가 증가하였다.

결 론: 염색 횟수의 증가는 단일 구조 지르코니아의 명도를 감소시키고, 황색 빛을 증가시킨다. 연마 처리는 명도를 감소시키고, 광택 처리는 명도를 감소시킬 뿐만 아니라 황색 빛을 증가시킨다. 삭제량의 증가는 명도를 감소시키고, 붉은 빛 및 푸른 빛을 증가시킨다. 삭제량의 증가는 투명도를 증가시킨다.

· **주요어:** 단일 구조 지르코니아, 염색액, 색상, 투명도, 표면 특성 변화
· **학번:** 2003-31121

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Effect of Various Surface Modifications on the Optical Properties of Dental Monolithic Zirconia Restorations

다양한 표면 변화가 치과용 단일 구조 지르코니아 수복물의 광학적 특성에 미치는 효과에 관한 연구

2014 년 2 월

서울대학교 대학원

치의과학과 치과보철학전공

김 희 경

Effect of Various Surface Modifications on the Optical Properties of Dental Monolithic Zirconia Restorations

2014

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*(Directed by Prof., **Jai-Bong Lee,** DDS, MSD, PhD)*

CONTENTS

ABSTRACT

Purpose. The purpose of this study was to investigate the effect of various surface modifications on the optical properties of monolithic zirconia restorations. Experiment I investigated the effect of the number of coloring liquid applications on the optical properties of monolithic zirconia. Experiment II investigated the effect of polishing and glazing on the color and translucency of monolithic zirconia. Experiment III investigated the effect of the amount of thickness reduction on the color and translucency of monolithic zirconia.

Material and Methods. BruxZir monolithic zirconia blocks and Tanaka ZirColor A2-coloring liquid were used for this study. Eighteen monolithic zirconia specimens (27.6 mm × 27.6 mm × 2.0 mm) were prepared for Experiment I and divided into 6 groups (n = 3) according to the number of coloring liquid applications (Group I to V) and Group O as a control. Color coordinates (CIE L^*, a^* and b^* value), color difference (ΔE^*_{ab}), translucency parameter (TP) and opalescence parameter (OP) were recorded. Forty-five monolithic zirconia specimens (16.3 mm × 16.4 mm × 2.0 mm) were prepared for Experiment II and divided into 5 groups according to the number of coloring liquid applications (Group I to V). Each group was divided into 3 subgroups according to the method of surface treatments (n = 3): N: no

treatment; P: polishing; G: glazing. CIE L^*, a^* and b^* value, ΔE^*_{ab} and TP were recorded. One-hundred sixty-five monolithic zirconia specimens (16.3 mm × 16.3 mm × 2.0 mm) were prepared for Experiment III and divided into 5 groups according to the number of coloring liquid applications (Group I to V). Each group was divided into 11 subgroups according to the amount of thickness reduction in 0.1-mm increments until final thickness was 1.0 mm (n = 3): Subgroup 0: no reduction; Subgroup 1: 0.1 mm reduction; Subgroup 2: 0.2 mm reduction; Subgroup 3: 0.3 mm reduction; Subgroup 4: 0.4 mm reduction; Subgroup 5: 0.5 mm reduction; Subgroup 6: 0.6 mm reduction; Subgroup 7: 0.7 mm reduction; Subgroup 8: 0.8 mm reduction; Subgroup 9: 0.9 mm reduction; Subgroup 10: 1.0 mm reduction. CIE L^*, a^* and b^* value, ΔE^*_{ab}, TP value were recorded. All measurements were performed on five different areas of each specimen according to CIELAB color space relative to the standard illuminant D65 on a reflection spectrophotometer. Data were analyzed using one-way ANOVA followed by multiple comparison Scheffé test, Pearson correlation and regression analysis (α = 0.05).

Results. According to the results of Experiment I, with the increase of the number of coloring liquid applications, CIE L^* and OP values decreased, while CIE b^* increased. Color differences among groups ranged from 1.3 to 15.7 ΔE^*_{ab} units. TP values were not significantly changed. Experiment II showed that there was a significant difference in CIE L^* between Subgroup

N and P, and in CIE b^* between Subgroup P and G in all groups. A perceptible color difference was obtained between Subgroup N and P in all groups and between Subgroup N and G in most groups ($\Delta E^*_{ab} > 3.7$). There was no significant difference in TP values between each subgroup in most groups. Experiment III showed that thickness reduction resulted in the decrease in CIE L^* and b^* values and the increase in a^* values. Perceptible color difference was obtained between Subgroup 0 and Subgroup 1 ($\Delta E^*_{ab} > 3.7$) in all groups. TP values generally increased with the increase of thickness reduction in all groups.

Conclusion. Within the limitations of this study, the following conclusions can be drawn. Increasing the number of coloring liquid applications results in a decrease of the lightness and an increase of the yellowish appearance of monolithic zirconia restorations. Polishing decreases the lightness and glazing also decreases the lightness, while it increases the yellowish appearance of monolithic zirconia restorations. Increasing thickness reduction decreases lightness and increases the reddish appearance and decrease yellowish appearance of monolithic zirconia restorations. Reduced thickness of monolithic zirconia produces more translucent monolithic zirconia restorations.

Keyword: Y-TZP ceramic; Color; Translucency; Dental Prosthesis Coloring; Surface modifications
Student number: 2003 – 31121

DECLARATION

I declare that no portion of the work referred to in the thesis has been submitted in support of an application for another degree or qualification of this or any other university or other institute of learning.

DEDICATION

To my family, Yong-Ho, Jin-Yung, Sung-Joon

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1. INTRODUCTION

Metal-ceramic restorations have been introduced since 1962 [1] and widely used in fixed dental prostheses for many decades as an esthetic restoration. For metal-ceramic restorations, light reflection from an opaque porcelain layer to mask the metal substrate resulted in opaque appearance and thereby, their use in high esthetic area was limited [2]. All-ceramic restorations without metal substrate induced more light transmission within the crown and accordingly, they improved the ability to reproduce the appearance of natural tooth [3]. However, brittleness was exhibited as a major drawback with being limited in a clinical use of all-ceramics [4]. Since the advent of high strength zirconia [5], zirconia-based material combined with CAD/CAM technology has broaden the range of their applications in dentistry. Due to its whitish and opaque color, it has to be veneered with feldspathic porcelain for more acceptable esthetic outcome, but cohesive failure of the veneering porcelain has been identified as a main complication [6, 7]. Several factors that reduce the cohesive failure of veneering porcelain have been studied. Fabricating optimized zirconia substructures [8], matching of thermal expansion between core and veneering ceramics [9], uniform thickness of cement layer [10], reduced thickness ratio of veneering porcelain [11], increased cooling rate [12] could reduce the incidence of cohesive veneering failure. Furthermore, in an attempt to reinforce the veneering porcelain, several trials such as high

strength CAD/CAM-fabrication of veneering porcelain [13], high strength heat-pressed ceramic [14], and "double veneering" technique [15] have been performed. Other approach to control the veneering failure could be a fabrication of monolithic zirconia which consists of a single zirconia material without any veneering. Nowadays the advantages of monolithic zirconia restorations with an increased mechanical stability make them possible to expand their clinical indications [16]. In addition to mechanical advantage of monolithic zirconia, this new approach also requires esthetic considerations.

Color and translucency can be determined from the transmission or reflection of the light that is absorbed, scattered, refracted and reflected [17]. Polycrystalline contents of zirconia induce maximum light scattering and diffuse reflection and thereby, result in opaque appearance to visible light [18]. Due to its inherent whitish and opaque appearance, the overall appearance of monolithic zirconia can be improved by surface modifications, such as coloring procedure, surface polishing and glazing. Unlike metal-ceramic restorations, monolithic zirconia can be colored in a pre-sintered state to match adjacent teeth.

There are common surface treatment methods for ceramic restorations, such as polishing and glazing. Sequential polishing procedures using various diamond points, rubber wheels, and abrasive pastes may give a luster to the

surface [19]. Glazing can be created either by firing a small coating of transparent glass onto the surface or by heating the restoration up to glazing temperature for 1 or 2 minutes to get shiny gloss surfaces [19]. Several studies [20-22] have compared glazing with different polishing techniques for ceramic restorations regarding surface texture. They demonstrated that polishing on feldspathic porcelain could be used as an alternative method for glazing. Other studies [23-25] investigated the effect of surface treatments on the color of porcelain restorations. According to these studies, surface treatments including polishing and glazing could affect the color of porcelain restorations. However, there have been no reported studies that investigated the effect of surface polishing and glazing on the color and translucency of monolithic zirconia.

The surface might also be modified in the adjustment procedures by the dentist to achieve an optimal occlusal contact. Grinding the ceramic surface with a diamond bur results in the thickness reduction of monolithic zirconia restorations. There have been several studies to investigate the importance of the ceramic thickness on overall color and translucency of ceramic restorations. However, those studies investigated the change of overall color of ceramic restorations with regard to masking effect of underlying substrate [26], core/veneer interaction [27, 28] and dentin porcelain overlying opaque substrate [2, 25, 29] based on metal-ceramic or all-ceramic restorations and

did not address the color change of material itself as a function of changes in thickness. Another previous study [30] determined the change of contrast ratio as a parameter for translucency measurements with the different thickness of all-ceramic core and veneering materials, respectively, to predict the translucency of multi-layered ceramic structures as a function of changes in thickness. However, there have been no reported studies regarding the change of color and translucency as a function of changes in thickness using monolithic zirconia which is a single-layered ceramic structure.

Any surface modifications of monolithic zirconia restorations might induce the shift of esthetic outcomes in terms of optical properties. In addition, there would be a significant implication of opalescence property concerning clinical application of monolithic zirconia due to its white and opaque appearance. The purpose of Experiment I was to investigate the effect of the number of coloring liquid applications on the color, translucency and opalescence of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in the optical properties between monolithic zirconia ceramics with the different number of coloring liquid applications. The purpose of Experiment II was to investigate the effect of polishing and glazing on the color and translucency of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in color and translucency parameters between monolithic zirconia ceramics

with the different surface treatments. The purpose of Experiment III was to evaluate the effect of the amount of thickness reduction on the color and translucency of monolithic zirconia. The null hypothesis to be tested was that there was no significant difference in color and translucency parameters between monolithic zirconia ceramics with different amounts of thickness reduction.

2. MATERIAL and METHODS

Monolithic zirconia (BruxZir, yttria-stabilized tetragonal zirconia polycrystal, Glidewell Laboratories, Newport Beach, CA, USA) was investigated in this study (Table 1). Tanaka ZirColor of A2 shade (Tanaka Dental, Skokie, IL, USA) was used as a coloring liquid (Table 1). It is designed to be brushed on and dried quickly with no drying time between each application and, thus no preheating is necessary before sintering.

2.1. Specimen preparation and optical properties measurement

2.1.1. ***Experiment I****. Effect of the number of coloring liquid applications on the optical properties of monolithic zirconia*

Eighteen square-shaped, pre-sintered zirconia block (34.0 mm × 34.0 mm × 2.7 mm) were fabricated using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a synthetic nylon fiber brush (No.156, Hwahong, Hwasung-si, Kyunggi-Do, Korea; Fig. 1). These specimens were divided into six groups (n = 3) according to the number of coloring liquid applications. The specimen with no application was used as a control.

- Group O (Control group): No application
- Group I: One time of application

Table 1. Materials investigated

Type	Brand name	Composition	Lot No.		Manufacturer
Monolithic zirconia block	BruxZir	Yttria-stabilized tetragonal zirconia polycrystal	Exp. I Exp. II Exp. III	B 186338 B 84942 B 105566 B 105583 B 105565 B 88712	Glidewell Laboratories, Newport Beach, CA, USA
Coloring liquid	Tanaka ZirColor (A2 shade)	(R)-p-mentha-1,8-diene, 50-75% Stoddard solvent, 10-25%	Exp. I Exp. II Exp. III	50297 50003 40127 40129	Tanaka Dental, Skokie, IL, USA

• Exp: Experiment

Fig. 1. (A) Tanaka ZirColor coloring liquid of A2 shade was used. (B) The coloring liquid was brushed on with a brush according to the manufacturer's recommendations.

- Group II: Two times of application
- Group III: Three times of application
- Group IV: Four times of application
- Group V: Five times of application

All specimens were then fired in a zirconia sintering furnace (LHT 0217, Nabertherm GmbH, Bahnhofstr, Germany). The sintering cycle was controlled as follows: The temperature was raised to 950°C for 1.5 hours and maintained for 2 hours, and then raised up to 1550°C for 1.5 hours and maintained for 3 hours. After sintering process, the shrinkage of specimens was *circa* 20%. The mean dimension of sintered specimens was 27.6 mm × 27.6 mm (Fig. 2), verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The thicknesses of specimens were 1.934 ± 0.059 mm. Colored surface of the specimen was neither grinding nor polishing after completion of sintering. All

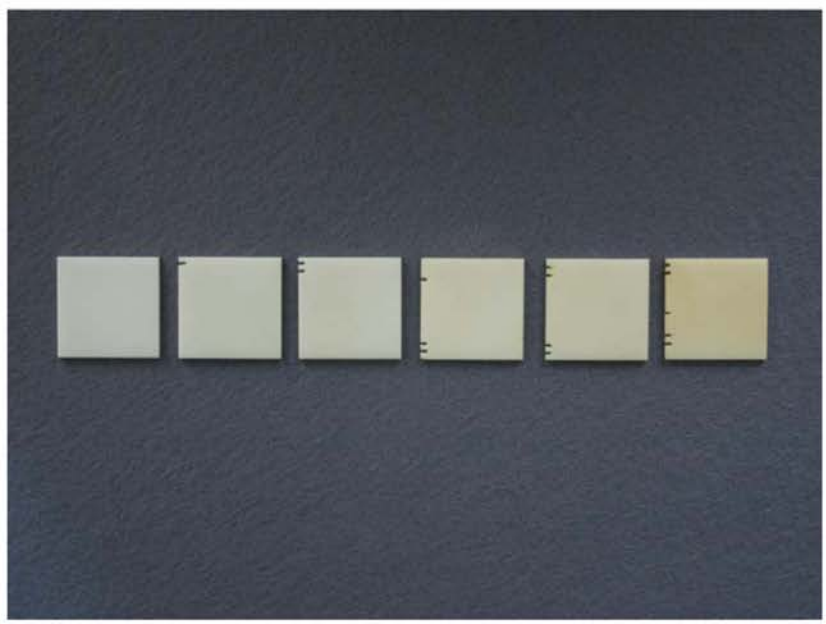

Fig. 2. Colored specimens of each group after sintering process. The number of slots on the left side indicated the number of coloring liquid applications (Experiment I).

specimens were ultrasonically cleaned in distilled water for 5 minutes before testing.

Color and spectral distribution were taken with a double-beam spectrophotometer (Cary 5000 UV-VIS-NIR Spectrophotometer, Agilent Technologies Inc., Santa Clara, CA, USA) (Fig. 3) using an integrating sphere attachment. The specular reflectance component was excluded (SCE mode) by gloss trap inserted. Relative reflectance data was recorded in the visible range from 380 to 780 nm at 5 nm intervals. Measurements were recorded in Commission Internationale de l'Eclairage (CIE) 1976 $L^*a^*b^*$ color space (CIELAB, Fig. 4) relative to the standard illuminant D65 in the reflectance mode over a white background (CIE $L^* = 99.9701$, $a^* = -0.0711$ and $b^* = 0.0499$) and a black background (CIE $L^* = 4.7487$, $a^* = -1.6749$ and $b^* = -1.5844$) and in the transmittance mode. The white standard was polytetrafluoroethylene (PTFE) plate (SRS-99-020, Spectralon® Reflectance Standards, Labsphere Inc., North Sutton, NH, USA) and the black background was a black tile (CM-A101B, Konica Minolta Optics Inc., Tokyo, Japan). The spectrophotometer was equipped with an integrating sphere of 150 mm diameter made with sintered PTFE. The geometry for the reflectance measurements was *8°:de* (eight degree: diffuse geometry, specular component excluded). Thus, CIE 1964 10° supplementary standard observer,

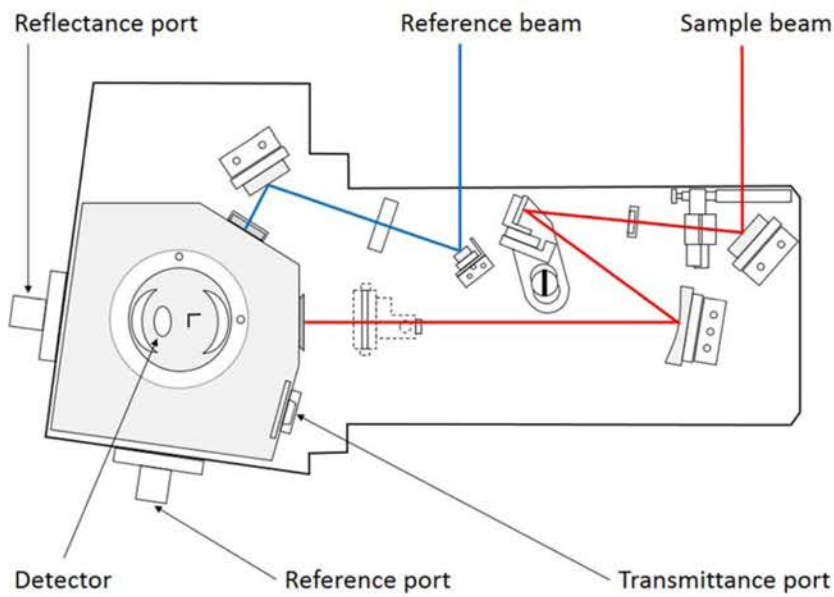

Fig. 3. A schematic view of double-beam spectrophotometer (Cary 5000 UV-VIS-NIR Spectrophotometer, Agilent Technologies Inc., Santa Clara, CA, USA).

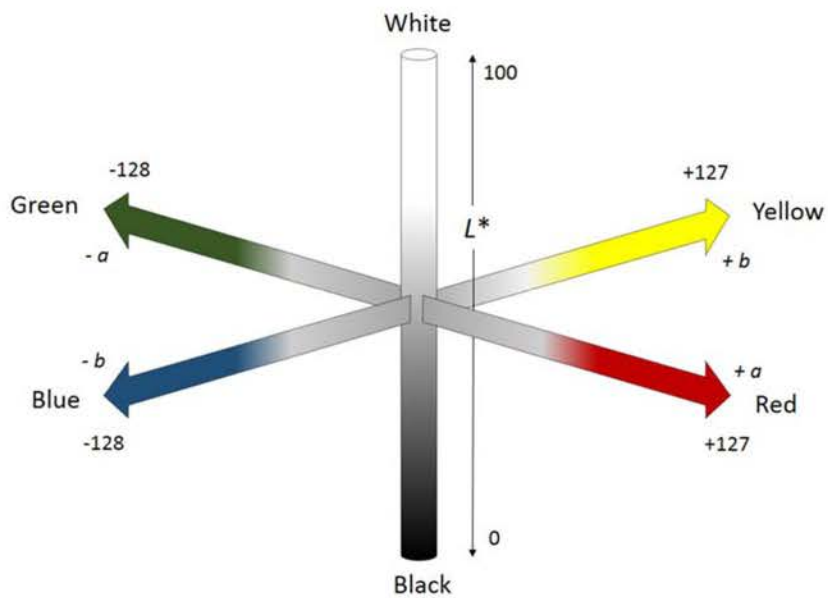

Fig. 4. CIE 1976 $L^*a^*b^*$ color space. The three coordinates of CIELAB represent the lightness of the color (L^*), its position between red and green (a^*) and its position between yellow and blue (b^*).

which is more modern and alternative to CIE 1931 2° standard observer, was selected. The aperture size was 19 mm in diameter for the reflectance measurement. For the transmittance measurement, opaque black polyvinyl chloride (PVC) plate supported measuring aperture to make the aperture size 10 mm × 15 mm, because the original aperture size of the instrument was 10 mm × 35 mm for the transmittance measurement. The specimens of 27.6 mm × 27.6 mm for this study provided adequate area for color measurement. The white PTFE standard was used for zero/base correction before reflectance color measurement.

Color coordinates, CIE L^*, a^* and b^*, were determined from the transmittance and reflectance data using a computer software (Cary WinUV Software, Agilent Technologies Inc., Santa Clara, CA, USA). Since the beam size was 1 mm × 5 mm, an effort was made not to overlap the beam on each measurement. Average L^*, a^* and b^* values were used to calculate CIE 1976 a,b (CIELAB) color difference, ΔE^*_{ab} of each group set using the following equation (Eq. 1) [31].

$$\Delta E^*_{ab} = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad \text{(Eq. 1)}$$

where, the L^* coordinate represents the lightness of an object, the a^* value represents the red or green chroma, and the b^*value represents the yellow or

blue chroma and ΔL^*, Δa^* and Δb^* indicate the differences between the CIE L^*, a^* and b^* color parameters of two specimen groups.

The translucency parameter (TP) was obtained by calculating the color difference of the specimen over the black and white backgrounds with the following equation (Eq. 2) [32].

$$TP = [(L_B^* - L_W^*)^2 + (a_B^* - a_W^*)^2 + (b_B^* - b_W^*)^2]^{1/2} \quad \text{(Eq. 2)}$$

where, subscript B refers to the color coordinates over a black background and the subscript W refers to those over a white background. TP value of zero corresponds to a completely opaque material. The greater the TP value, the higher the actual translucency of the material.

The opalescence parameter (OP) was calculated as the difference in yellow-blue and red-green coordinates between the transmitted and reflected colors using the following equation (Eq. 3) [33].

$$OP = [(\text{CIE } a_T^* - \text{CIE } a_R^*)^2 + (\text{CIE } b_T^* - \text{CIE } b_R^*)^2]^{1/2} \quad \text{(Eq. 3)}$$

where, subscript T refers to the transmitted color and subscript R refers to the reflected color over a black background. All measurements were performed

on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each group were obtained.

2.1.2. ***Experiment II****. Effect of polishing and glazing on the color and translucency of monolithic zirconia*

Forty-five square-shaped, pre-sintered zirconia blocks (20.0 mm × 20.0 mm × 2.7 mm) were prepared using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a brush (2850-B, Babara, Kobe, Japan). These specimens were divided into five groups according to the number of coloring liquid applications.

- Group I: One time of application
- Group II: Two times of application
- Group III: Three times of application
- Group IV: Four times of application
- Group V: Five times of application

All specimens were then sintered in a zirconia sintering furnace (LHT 0217, Nabertherm GmbH, Bahnhofstr, Germany). The sintering cycle was controlled following the protocol of Experiment I. After sintering process,

shrinkage of specimens was *circa* 20%. The mean dimension of sintered specimens was 16.3 mm × 16.4 mm (Fig. 5), verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The specimen thicknesses were 1.990 ± 0.025 mm. Nine specimens of each group were assigned to three subgroups according to the surface treatment (n = 3).

- Subgroup *-N (Control group): No treatment
- Subgroup *-P: Polishing
- Subgroup *-G: Glazing

*: I, II, III, IV or V

In Subgroup P, specimens were polished with a sequence of three diamond-impregnated silicone discs for 60 seconds each. Then, specimens were polished using a felt wheel with diamond polishing paste for 60 seconds each

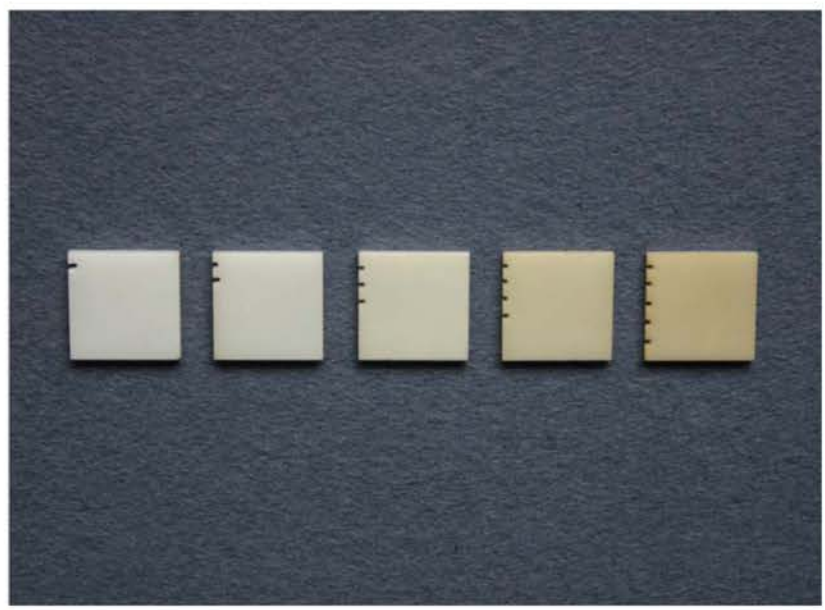

Fig. 5. Colored specimens of each group after sintering process. The number of slots on the left side indicated the number of coloring liquid applications (Experiment II).

consecutively (Fig. 6). Details of polishing instruments were described in Table 2.

In Subgroup G, specimens were glazed in vacuum in a ceramic furnace (Austromat 3001, DEKEMA Dental-Keramiköfen GmbH, Freilassing, Germany) using a glazing paste (Fig. 7, Table 3) for each, following the protocol: The temperature was raised up to 950°C at the firing rate of 30°C/min, and maintained for 30 seconds, and then cooling down to 300°C at 15°C/min.

Subgroup N indicating no surface treatment served as a control. All specimens were ultrasonically cleaned in distilled water for 5 minutes before testing.

Colors were measured according to CIELAB color space in the reflectance mode relative to the standard illuminant D65 on a reflection spectrophotometer (Fig. 8), which was equipped with an integrating sphere. Illuminating and viewing configurations of this instrument were *de:8°* geometry and the 10° CIE 1964 supplementary standard colorimetric observer was selected. The aperture diameter of the measuring port of the spectrophotometer (Target Mask CM-A 121, Minolta, Osaka, Japan) was 3 mm. White calibrating plate (CM-A120, Minolta, Osaka, Japan) was

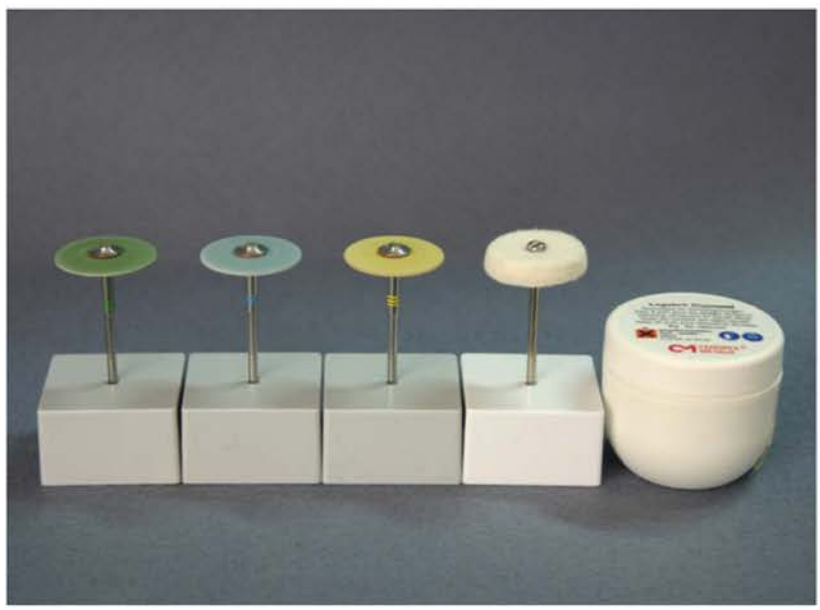

Fig. 6. Three diamond impregnated silicone discs (green: coarse grit, blue: medium-coarse grit, yellow: super-fine grit, Edenta AG, Au SG, Switzerland) and felt wheel (Super-Snap Buff, Shofu Inc., Kyoto, Japan) with diamond polishing paste (LegabrilDiamond, Metalor Dental AG, Biel/Bienne, Switzerland) (Experiment II).

Table 2. Polishing instruments used

Instruments	Lot No.	Grit/Contents	RPM	Manufacturer
CeraGloss 310 HP (Green)	P04.002	Coarse grit/ diamond particles	5,000	Edenta AG, Au SG, Switzerland
CeraGloss 3010 HP (Blue)	R09.003	Medium-coarse grit/ diamond particles	5,000	
CeraGloss 30010 HP (Yellow)	T02.001	Super-fine grit/ diamond particles	5,000	
LegabrilDiamond	08052307	diamond paste		Metalor Dental AG, Biel/Bienne, Switzerland

• RPM: Revolutions per minute

Fig. 7. Glaze material in paste form (IPS e.max Ceram Glaze, Ivoclar Vivadent AG, Schaan, Liechtenstein). The glaze paste was mixed with the IPS e.max Ceram Glaze and Stain Liquid until the desired consistency was reached (Experiment II).

Table 3. Glazing material used

Type	Brand name	Composition (%)	Lot No.	Manufacturer
Glazing paste	IPS e.max Ceram Glaze	SiO_2, 61.0 – 68.0 Al_2O_3, 5.0 – 8.0 Na_2O, 5.0 – 8.0 K_2O, 5.0 – 8.0 ZnO, 2.0 – 4.0 Other oxides, 3.5 – 17.0 Pigments, 0.0 – 1.0 Glycerine, 20.0 – 25.0 1,3-Butandiol, 15.0 – 20.0	R85911	Ivoclar Vivadent AG, Schaan, Liechtenstein

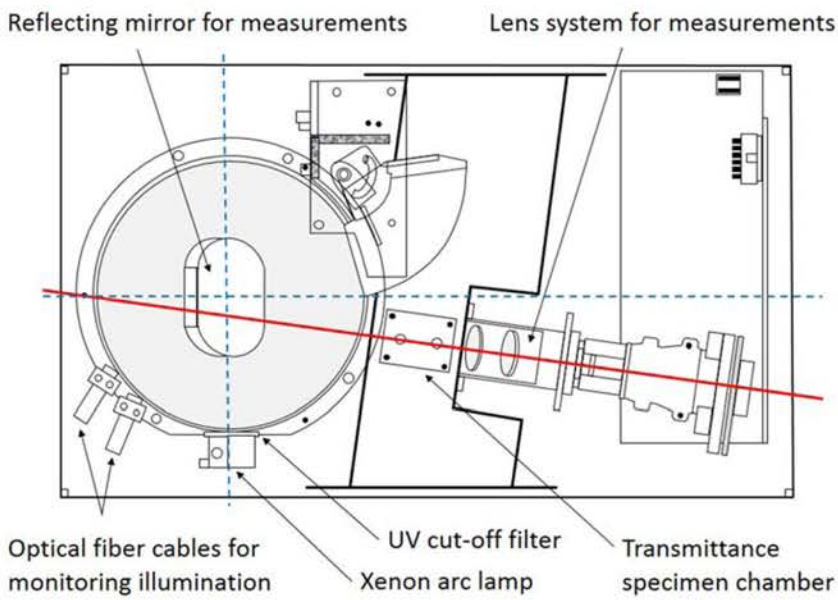

Fig. 8. A schematic view of reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan).

performing for the white calibration standard for reflectance measurements, and zero calibration box (CM-A 124, Minolta, Osaka, Japan) was for zero calibration for reflectance measurements. CIE L^*, a^* and b^* values were measured over a zero calibrating box (CM-A 124, Minolta, Osaka, Japan, CIE $L^* = 0.1099$, $a^* = 0.2107$ and $b^* = -0.4292$) with SCE under ultraviolet light excluded conditions and spectral reflectance over the white background (CM-A120, Minolta, Osaka, Japan, CIE $L^* = 96.6880$, $a^* = -0.1755$ and $b^* = -0.1236$) were measured in the range of visible wavelengths of 400 to 700 nm at 10 nm intervals. A drop of distilled water whose refractive index is 1.7, was placed between specimen and the background for better optical contact [28]. All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly.

Color coordinates, CIE L^*, a^* and b^*, were determined from the reflectance data using a computer software (Spectra-Magic version 1.01, Minolta, Osaka, Japan). Average L^*, a^* and b^* values were used to calculate CIE 1976 a,b (CIELAB) color difference, ΔE^*_{ab} of each group set using Eq. 1.

TP value of each subgroup within groups was calculated using Eq. 2. Spectral transmittance was recorded at 10 nm intervals in the range of 400 to 700 nm in the transmittance mode under ultraviolet light excluded conditions. The

aperture size was 9.56 mm in diameter which was made of an opaque black graphite plate with a central window, because the original aperture size of the instrument was 25 mm in diameter for the transmittance measurement. From the data, percent transmittance was calculated.

All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each subgroup in each group were obtained.

2.1.3. ***Experiment III****. Effect of the amount of thickness reduction on the color and translucency of monolithic zirconia*

One-hundred sixty-five square-shaped, pre-sintered zirconia blocks (20.0 mm × 20.0 mm × 2.7 mm) were prepared for Experiment III using a cutting machine (618 slicer, Harig, Niles, IL, USA). The coloring liquid was applied according to the manufacturer's recommendations with a brush (2850-B, Babara, Kobe, Japan). These specimens were divided into five groups according to the number of coloring liquid applications.

- Group I: One time of application
- Group II: Two times of application
- Group III: Three times of application

- Group IV: Four times of application
- Group V: Five times of application

All specimens were then sintered in a zirconia sintering furnace (Austromat *ba*SiC®, DEKEMA Dental-Keramiköfen GmbH, Freilassing, Germany). The sintering cycle was controlled as follows: The temperature was raised to 950°C for 63 minutes and maintained for 10 minutes, and then raised up to 1500°C for 55 minutes and maintained for 2 hours. After sintering process, the shrinkage of specimens was *circa* 20%. The mean size of sintered specimens was 16.3 mm × 16.3 mm, verified with a Vernier caliper (Mitutoyo, Tokyo, Japan).

The grinding procedure was performed on the opposite side of colored surface of each specimen to adjust the final thickness to 2.0 mm by the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea). Final thickness was checked with a digital height gauge (Digimicro ME-50HA, Nikon Corp, Tokyo, Japan) with the accuracy of 1 μm on five different sites (center and each corner of specimen) of each specimen. The specimen thicknesses were 1.997 ± 0.017 mm. Thirty-three specimens of each group were assigned to eleven subgroups according to the amount of thickness reduction (n = 3).

- Subgroup *-0 (Control group): No reduction
- Subgroup *-1: 0.1 mm reduction
- Subgroup *-2: 0.2 mm reduction
- Subgroup *-3: 0.3 mm reduction
- Subgroup *-4: 0.4 mm reduction
- Subgroup *-5: 0.5 mm reduction
- Subgroup *-6: 0.6 mm reduction
- Subgroup *-7: 0.7 mm reduction
- Subgroup *-8: 0.8 mm reduction
- Subgroup *-9: 0.9 mm reduction
- Subgroup *-10: 1.0 mm reduction

*: I, II, III, IV or V

Thickness reduction of each subgroup was performed on the colored surface using the horizontal grinding machine (HRG-150, AM Technology, Asan, Chungcheongnam-do, Korea) according to the protocol, respectively. Means and standard deviations of thickness of each subgroup are shown in Table 4. Colors were measured according to CIELAB color space relative to the standard illuminant D65 on a reflection spectrophotometer (CM-3500d, Minolta, Osaka, Japan), which was equipped with an integrating sphere. Illuminating and viewing configurations was followed by the protocol of

Table 4. Means and standard deviations in parentheses for the thickness of each subgroup

Subgroup	Thickness (mm)
0	2.00 (0.017)
1	1.91 (0.003)
2	1.81 (0.003)
3	1.71 (0.013)
4	1.61 (0.003)
5	1.51 (0.003)
6	1.41 (0.002)
7	1.31 (0.003)
8	1.21 (0.002)
9	1.11 (0.016)
10	1.01 (0.002)

Experiment II. Each of L^*, a^* and b^* values was measured over a zero calibrating box (CM-A 124, Minolta, Osaka, Japan, $L^* = 0.1099$, $a^* = 0.2107$ and $b^* = -0.4292$) and the white background (CM-A120, Minolta, Osaka, Japan, $L^* = 96.6880$, $a^* = -0.1755$ and $b^* = -0.1236$) in the reflectance mode with SCE under ultraviolet light excluded conditions at 10 nm intervals in the range of visible wavelengths of 400 to 700 nm. The aperture diameter of the reflectance measurement of the spectrophotometer (Target Mask CM-A 121, Minolta, Osaka, Japan) was 3 mm. Calibration of spectrophotometer was performed before measurements. A drop of distilled water was placed between specimen and the background for better optical contact. Spectral reflectance over the white background was also recorded at 10 nm intervals in the range of 400 to 700 nm.

Color coordinates, L^*, a^* and b^*, were determined from the reflectance data using a computer software (Spectra-Magic version 1.01, Minolta, Osaka, Japan). Average L^*, a^* and b^* values over the zero calibration box were used to calculate color difference, ΔE^*_{ab} of each group set using Eq. 1. TP value was obtained by calculating the color difference of the specimen over the black calibration box and white background using Eq. 2. Spectral transmittance was recorded at 10 nm intervals in the range of 400 to 700 nm in the transmittance mode under ultraviolet light excluded conditions. The aperture size was 9.56 mm in diameter which was made of an opaque black

graphite plate with a central window, because the original aperture size of the instrument was 25 mm in diameter for the transmittance measurement. From the data, percent transmittance was calculated.

All measurements were performed on five different areas of each specimen including the center of specimen by moving it to each quadrant direction slightly. Thus, fifteen measurement data per each subgroup in each group were obtained.

2.2. Statistical analysis

SPSS software (version 20.0, SPSS Inc., Chicago, IL, USA) was used for statistical analyses and the probability level for statistical significance was set at $\alpha = 0.05$. One-way analysis of variance (ANOVA) and multiple comparison Scheffé test were performed to determine whether there were any significant differences in each parameter between the groups or subgroups.

For Experiment I, each of CIE L^*, a^*, b^* value, TP and OP value after coloring was used as a dependent variable, and the number of coloring liquid applications was used as an independent variable. The correlation between the number of coloring liquid applications and CIE L^*, a^*, b^* value, TP and OP value, was found out by using the Pearson correlation coefficient. The

linear regression was fitted to analyze the influences of the number of coloring liquid applications on CIE L^*, a^*, b^* value, TP and OP value.

For Experiment II, each of CIE L^*, a^*, b^* value and TP value was used as a dependent variable, and each surface treatment was used as an independent variable. Correlation between CIE L^*, a^* and b^* values of each surface treatment and the number of coloring liquid applications was found out by using the Pearson correlation coefficient. The linear regression was fitted to analyze the influences of the number of coloring liquid applications on CIE L^*, a^* and b^* values of each surface treatment.

For Experiment III, each of L^*, a^*, b^* value and TP value was used as a dependent variable, and the amount of thickness reduction was used as an independent variable. Correlation between the amount of thickness reduction and L^*, a^*, b^* value and TP value was found out by using Pearson correlation coefficient. The linear regression was fitted to analyze the influence of the amount of thickness reduction on L^*, a^*, b^* value and TP value.

3. RESULTS

3.1. Experiment I. Effect of the number of coloring liquid applications on the optical properties of monolithic zirconia

Spectral distributions and color coordinates were measured in the reflectance mode over the white and black backgrounds, and in the transmittance mode. Each spectral distribution in the different mode exhibited different spectral behavior (Fig. 9, 10 and 11). Means and standard deviations for CIE L^*, a^* and b^* values of each group are listed in Table 5. CIE L^* and b^* value were significantly influenced by the number of coloring liquid applications. An increase in the number of coloring liquid applications produced a decrease in the L^* value resulting in darker specimens and an increase in the b^* value resulting in more yellowish specimens, in the reflectance mode over a white and black background and in the transmittance mode. Fig. 12 showed changes in the CIE L^* and b^* values with the increase of the number of coloring liquid applications based on the reflected light over a white background. There was no significant difference in a^* value of reflectance mode over the white and black backgrounds among Group I, II and III. With regard to a^* value in transmittance mode, there was no significant difference among groups except for Group O (Table 5).

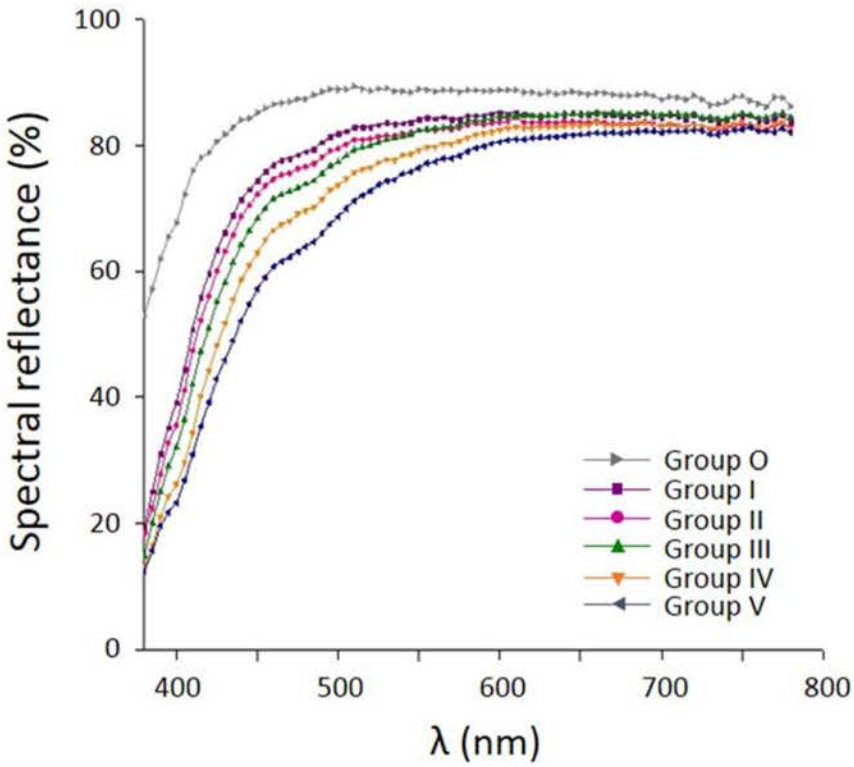

Fig. 9. Spectral reflectance over a white background of each group.

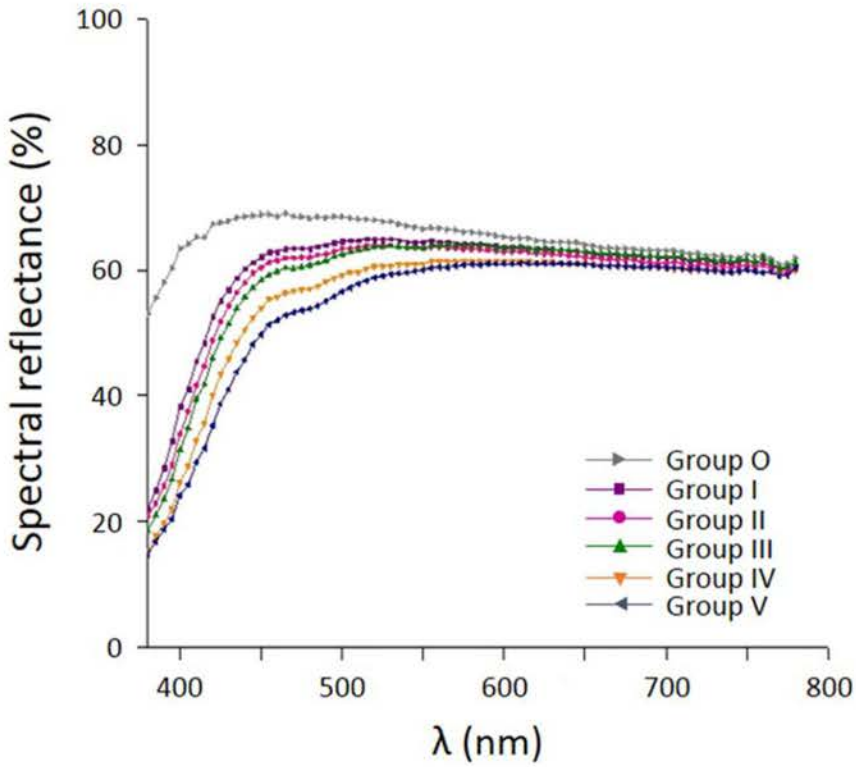

Fig. 10. Spectral reflectance over a black background of each group.

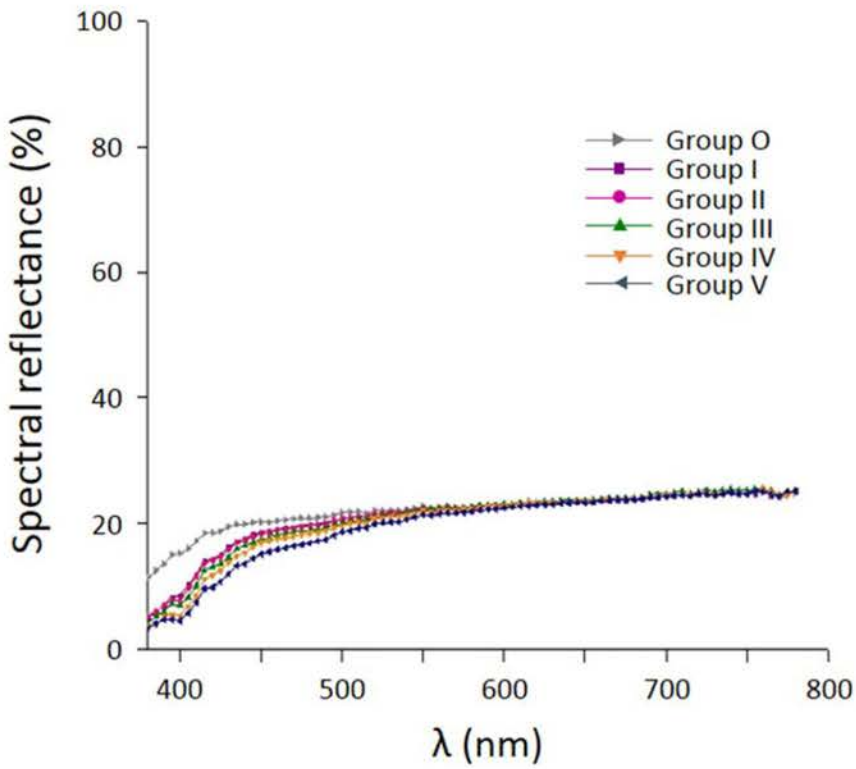

Fig. 11. Spectral transmittance of each group.

Table 5. Means (standard deviations) for CIE L^*, a^* and b^* values of each group

Group	Reflectance mode over the white background			Reflectance mode over the black background			Transmittance mode		
	L^*	a^*	b^*	L^*	a^*	b^*	L^*	a^*	b^*
O	95.38	-1.45[c]	2.95	85.52	-1.39	-1.37	54.22[a]	0.09	4.04
	(0.37)	(0.27)	(0.27)	(0.33)	(0.94)	(0.08)	(0.82)	(0.14)	(0.18)
I	93.17	-2.26[a]	8.36[a]	84.17	-2.38[a]	3.43[a]	53.76[a,b]	-0.37[a]	7.39[a]
	(0.61)	(0.12)	(1.03)	(0.34)	(0.08)	(0.97)	(0.53)	(0.15)	(0.77)
II	92.33[a]	-2.27[a]	9.35[a]	83.47[a]	-2.43[a,b]	4.10[a]	53.86[a,b]	-0.47[a]	7.80[a]
	(0.56)	(0.19)	(1.05)	(0.31)	(0.10)	(0.95)	(0.45)	(0.13)	(0.56)
III	92.14[a]	-2.10[a,b]	12.20	83.63[a]	-2.51[a,b]	6.26	53.69[a,b]	-0.29[a]	9.16
	(0.60)	(0.24)	(1.25)	(0.32)	(0.14)	(0.95)	(0.43)	(0.18)	(0.72)
IV	90.84	-1.98[b]	14.57	82.19	-2.60[c]	8.17	53.29[b]	-0.37[a]	10.31
	(0.39)	(0.20)	(0.79)	(0.17)	(0.15)	(0.39)	(0.34)	(0.17)	(0.54)
V	89.48	-1.49[c]	17.50	81.50	-2.55[b,c]	10.99	52.45	-0.28[a]	13.05
	(0.58)	(0.21)	(0.62)	(0.37)	(0.20)	(0.41)	(0.49)	(0.14)	(0.80)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

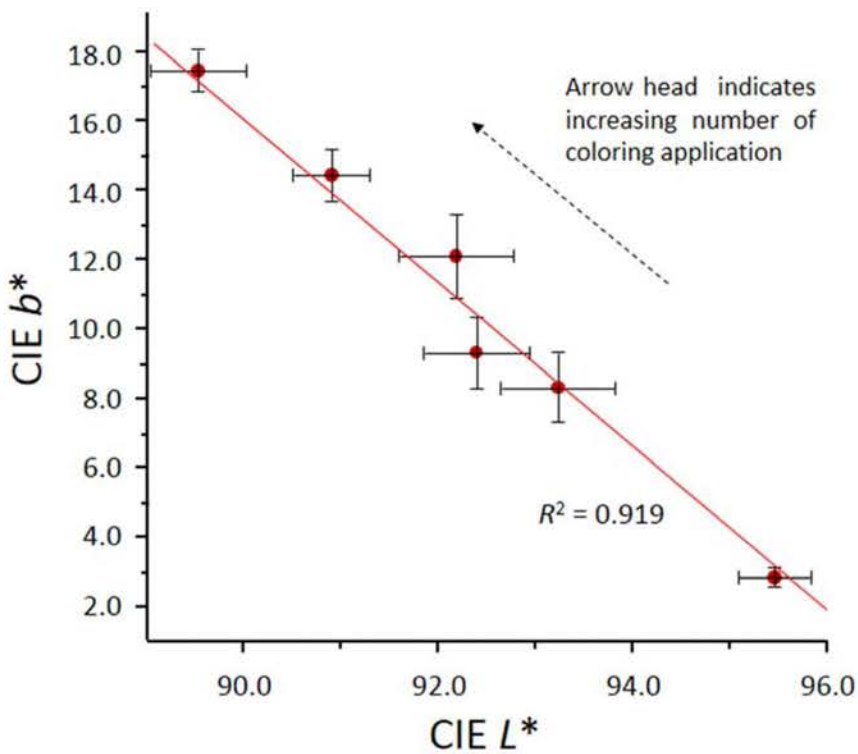

Fig. 12. Changes in the CIE L^* and b^* values with the increase of the number of coloring liquid applications based on the reflected light over a white background (Experiment I).

The color difference between each pair of groups was in the range from 1.3 to 15.7 ΔE^*_{ab} units (Table 6). The mean L^*, a^* and b^* values of each group over the white background in the reflectance mode were used to calculate the ΔE^*_{ab} between groups. The highest ΔE^*_{ab} value was 15.7 between Group O and V, while the lowest one was 1.3 between Group I and II. A perceptible color difference in a clinical setting ($\Delta E^*_{ab} > 3.7$) was obtained for all colored groups when compared with Group O. Color differences between two subsequent groups, such as I and II, II and III, III and IV, and IV and V, were not clinically perceptible ($\Delta E^*_{ab} < 3.7$).

Means and standard deviations of TP for each group are listed in Table 7. The statistical analyses showed no significant difference in TP value after the coloring procedure.

Means and standard deviations for the differences between the reflected colors over the black background and transmitted colors of each group are listed in Table 8. The OP values and Δb^* (the difference of CIE b^* value between transmitted color and reflected color) decreased as the number of coloring liquid applications increased. There was a negative correlation between the number of coloring liquid applications and OP value, indicating the Pearson correlation coefficients (r) to be −0.837. From a linear regression analysis, the coefficient of determination (R^2) was 0.701 (Fig. 13).

Table 6. Color differences between each group set

Group set	ΔE^*_{ab}
I-II	1.30
III-IV	2.71
II-III	2.85
IV-V	3.27
I-III	3.97
II-IV	5.43
O-I	5.90
III-V	5.96
I-IV	6.63
O-II	7.14
II-V	8.66
O-III	9.82
I-V	9.88
O-IV	12.48
O-V	15.70

• Group sets are arranged in ascending order of their ΔE^*_{ab} values.

Table 7. Means and standard deviations in parentheses for translucency parameter of each group

Group	TP
O	10.77[a] (0.47)
I	10.29[a] (0.41)
II	10.32[a] (0.41)
III	10.39[a] (0.43)
IV	10.80[a] (0.45)
V	10.39[a] (0.58)

- Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).
- TP denotes translucency parameter.

Table 8. Means and standard deviations in parentheses for the differences between the reflected and transmitted colors of each group

Group	OP	ΔE^*_{ab}	ΔL^*	Δa^*	Δb^*
O	5.61	31.81	31.31	1.48	5.41
	(0.20)	(1.08)	(1.08)	(0.11)	(0.20)
I	4.46[a]	30.74[a]	30.41[a]	2.01[a,b]	3.96[a]
	(0.54)	(0.56)	(0.55)	(0.16)	(0.64)
II	4.21[a,b]	29.91[a,b]	29.60[a,b]	1.95[a]	3.69[a,b]
	(0.71)	(0.75)	(0.69)	(0.17)	(0.87)
III	3.70[b,c]	30.17[a]	29.94[a]	2.22[b,c]	2.90[b,c]
	(0.57)	(0.33)	(0.32)	(0.18)	(0.81)
IV	3.14[c]	29.07[b]	28.89[b]	2.23[c]	2.14[c,d]
	(0.38)	(0.39)	(0.36)	(0.24)	(0.61)
V	3.11[c]	29.22[b]	29.05[b]	2.27[c]	2.06[d]
	(0.44)	(0.67)	(0.68)	(0.18)	(0.70)

- Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).
- OP denotes opalescence parameter.
- ΔE^*_{ab}, ΔL^*, Δa^* and Δb^* denote the differences of L^*, a^* and b^* values between transmitted and reflected color.

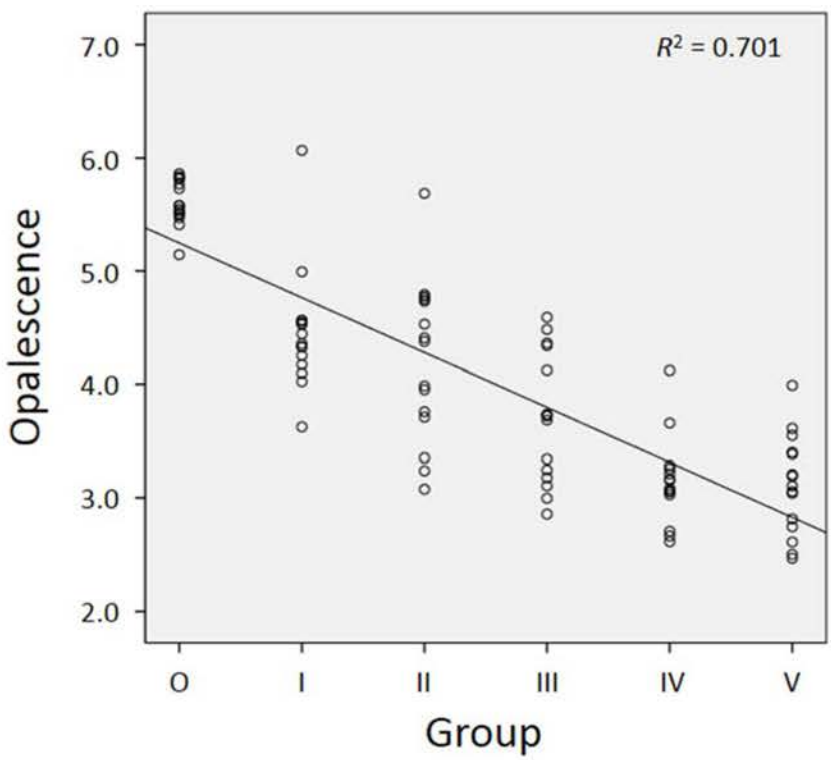

Fig. 13. Linear regression of opalescence values as a function of the number of coloring liquid applications (Experiment I).

Correlations between the number of coloring liquid applications and CIE L^*, a^* or b^* values were identified. There was a significant correlation between the number of coloring liquid applications and CIE L^* or b^* value indicating r value to be −0.937 or 0.968, R^2 to be 0.878 or 0.938, respectively (Fig. 14 and 15), whereas no significant correlation was found between the number of coloring liquid applications and CIE a^* value.

Correlations between OP and Δb^*, ΔE^*_{ab}, ΔL^* or Δa^* were identified. Between OP and Δb^*, r value was 0.991 and a regression equation, OP = $0.74\Delta b^* + 1.56$ ($R^2 = 0.982$) was calculated (Fig. 16). Based on the results of the present study, there were also significant correlations between OP and ΔE^*_{ab} ($r = 0.788$, $R^2 = 0.621$, Fig. 17), and OP and ΔL^* ($r = 0.736$, $R^2 = 0.541$, Fig. 18), but their correlations were lower than those between OP and Δb^*. However, there was a negative correlation between OP and Δa^* ($r = -0.782$, $R^2 = 0.612$, Fig. 19).

3.2. Experiment II. Effect of polishing and glazing on the color and translucency of monolithic zirconia

Means and standard deviations of L^*, a^* and b^* values over a zero calibrating box in the reflectance mode within each group as a function of surface treatment are listed in Table 9. As for L^* value, there was a significant difference between Subgroup N and P in each group, and between Subgroup

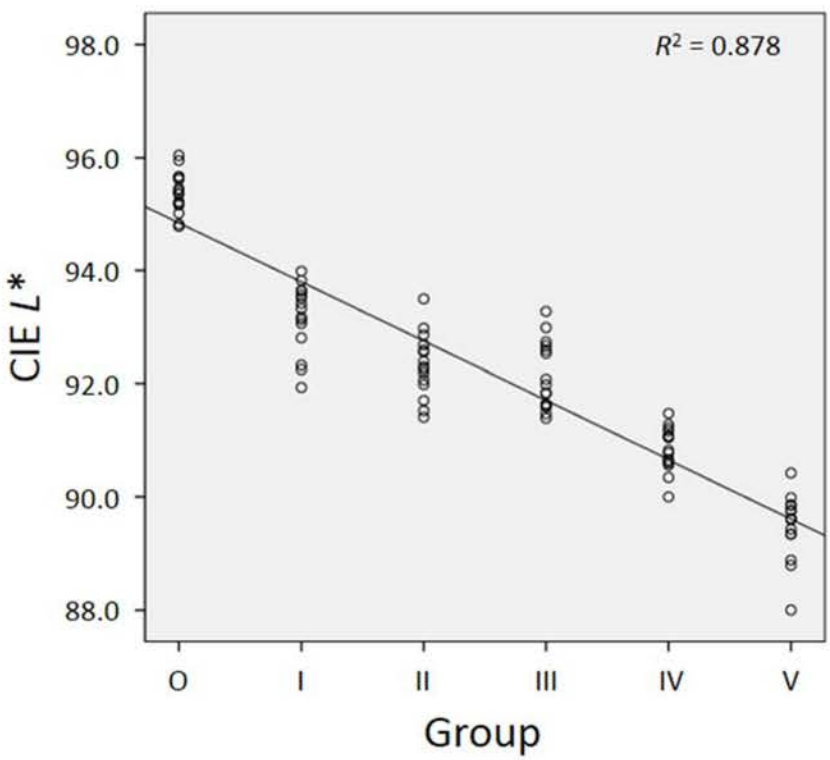

Fig. 14. Linear regression of CIE L^* values over a white background in the reflectance mode as a function of the number of coloring liquid applications (Experiment I).

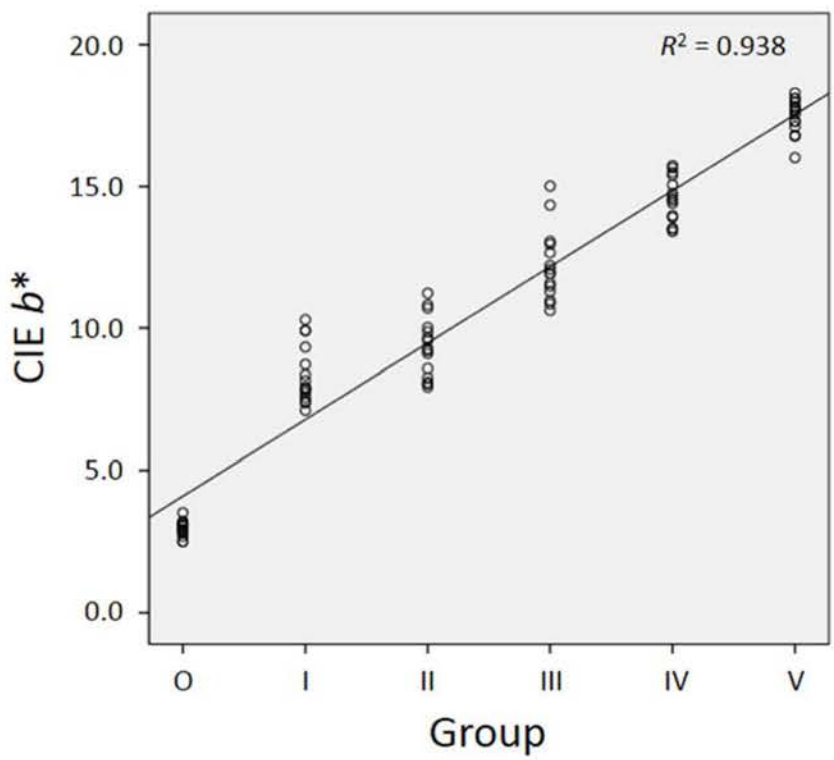

Fig. 15. Linear regression of CIE b^* values over a white background in the reflectance mode as a function of the number of coloring liquid applications (Experiment I).

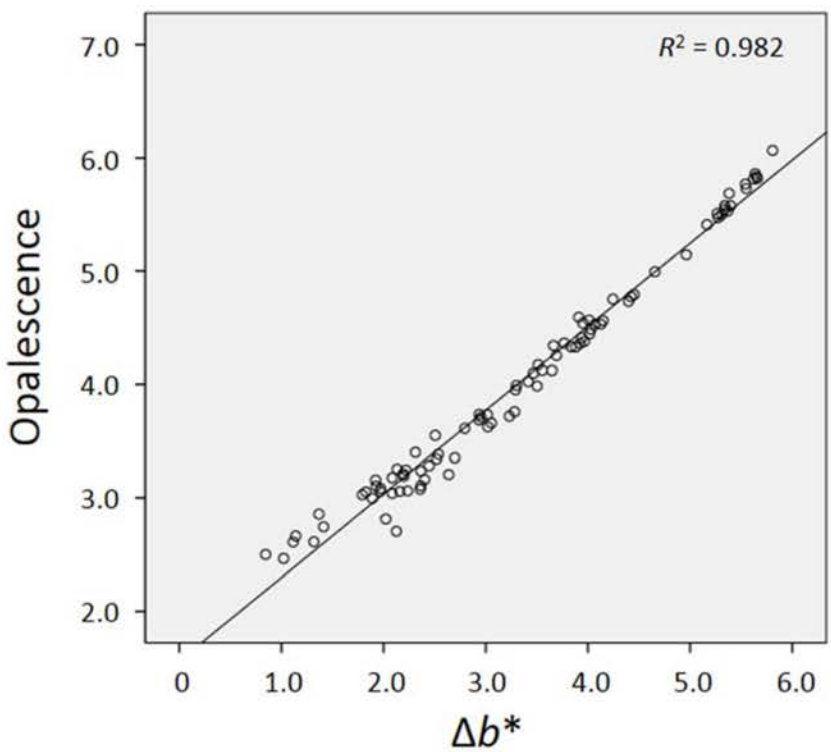

Fig. 16. Linear regression of opalescence values as a function of Δ*b** values (Experiment I).

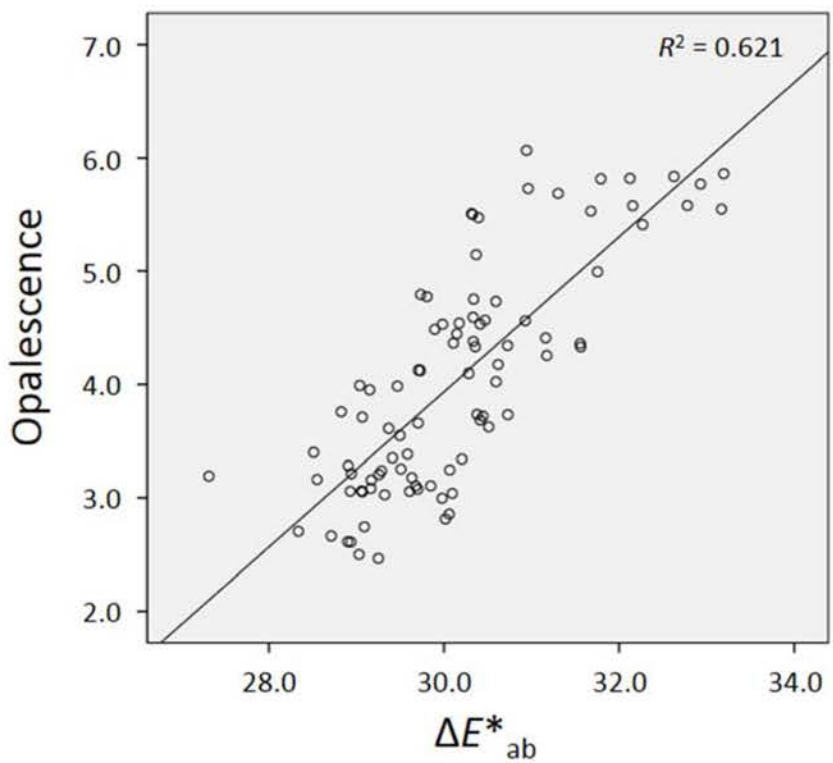

Fig. 17. Linear regression of opalescence values as a function of ΔE^*_{ab} values (Experiment I).

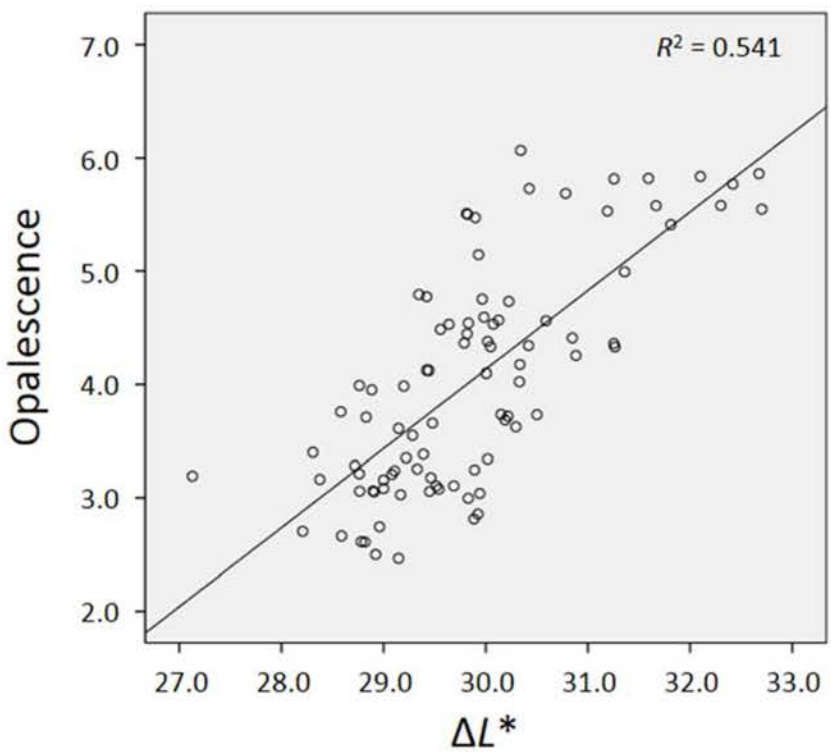

Fig. 18. Linear regression of opalescence values as a function of ΔL^* values (Experiment I).

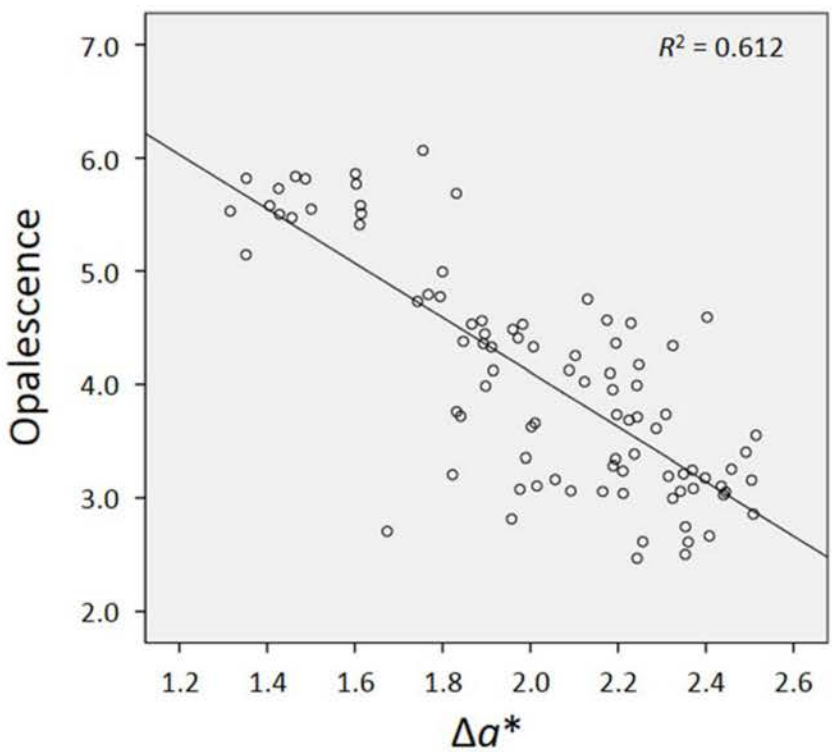

Fig. 19. Linear regression of opalescence values as a function of Δa^* values (Experiment I).

Table 9. Means (standard deviations) for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within each group as a function of surface treatment

	Surface treatment	Group				
		I	II	III	IV	V
L^*	N	73.49[a] (3.52)	70.49 (3.33)	70.92 (0.41)	71.17 (2.22)	66.37 (1.94)
	P	67.57[b] (3.82)	63.35 (2.47)	65.94[a] (2.87)	61.42 (1.45)	61.31[a] (3.07)
	G	70.61[a,b] (4.44)	67.04 (3.20)	66.97[a] (3.64)	64.58 (2.33)	61.27[a] (2.77)
a^*	N	-1.75[c] (0.14)	-2.12 (0.20)	-2.68[b] (0.11)	-2.33 (0.15)	-1.94[b] (0.28)
	P	-2.02[c,d] (0.29)	-2.66[a] (0.18)	-2.83[b,c] (0.19)	-3.07 (0.15)	-2.44 (0.33)
	G	-2.10[d] (0.45)	-2.67[a] (0.34)	-2.90[c] (0.21)	-2.90 (0.25)	-2.13[b] (0.28)
b^*	N	-2.87[e] (0.75)	-1.80[b] (0.68)	2.03 (0.73)	3.83[a] (0.73)	9.10[c] (1.02)
	P	-3.65[f] (0.24)	-1.71[b] (0.73)	3.47 (1.01)	3.43[a] (0.47)	9.80[c] (1.51)
	G	-2.72[e] (0.15)	-0.61 (0.70)	4.39 (0.70)	5.06 (0.87)	12.13 (0.70)

• Means with the same superscript letter in each group column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

N and G in each group except Group I. There was no significant difference between Subgroup P and G in Group I, III and V. As for a^* value, there was a significant difference between Subgroup N and P in Group II, IV and V and between Subgroup P and G in Group IV and V. There was a significant difference between Subgroup N and G except Group V. As for b^* value, there was no significant difference between Subgroup N and P in Group II, IV and V. There was a significant difference between Subgroup P and G in each group, and between Subgroup N and G in each group except Group I.

Fig. 20 to 27 show the spectral reflectance against the white background of specimens within groups or subgroups. Each surface treatment exhibited similar spectral behavior through the entire spectrum in the range of 400 to 700 nm, but the values of spectral reflectance in Subgroup P and G were generally lower than those in Subgroup N (Fig. 20 to 24). There was no significant difference between Subgroup P and G in Group I, III and V (Fig. 20, 22 and 24). There was a significant difference between each surface treatment in Group I, II and IV (Fig 20, 21 and 23), representing the highest value in Subgroup N and the lowest value in Subgroup P except for the short wavelengths of *circa* 400 nm ($\alpha = 0.05$). Fig. 25 to 27 presented the spectral reflectance of each group within Subgroup N, P and G. There was a gradually decreasing tendency of spectral reflectance as the number of coloring liquid

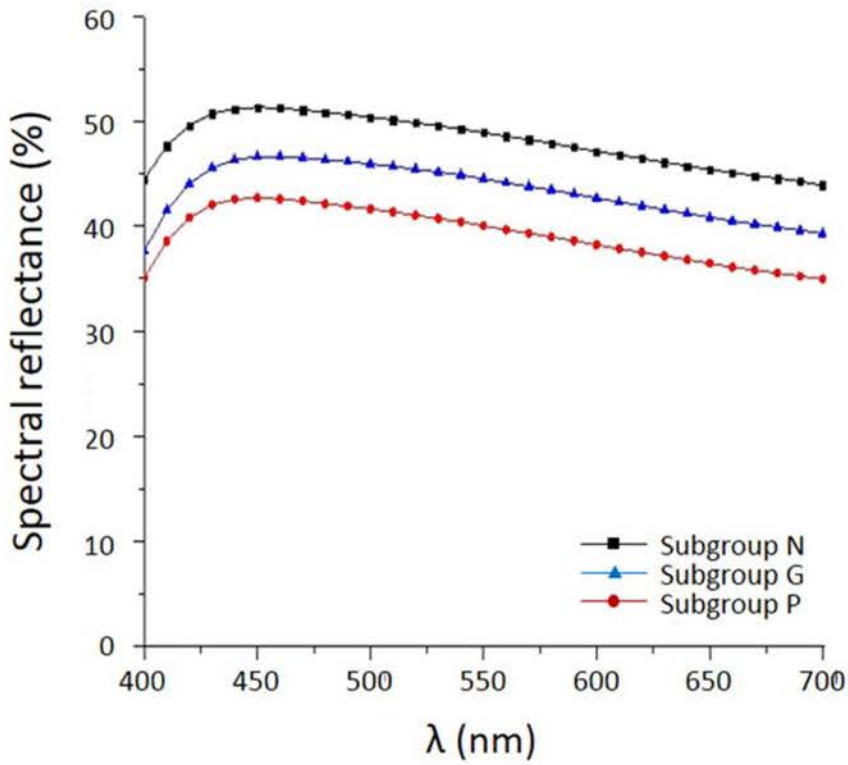

Fig. 20. Spectral reflectance of each subgroup in Group I against white background (Experiment II).

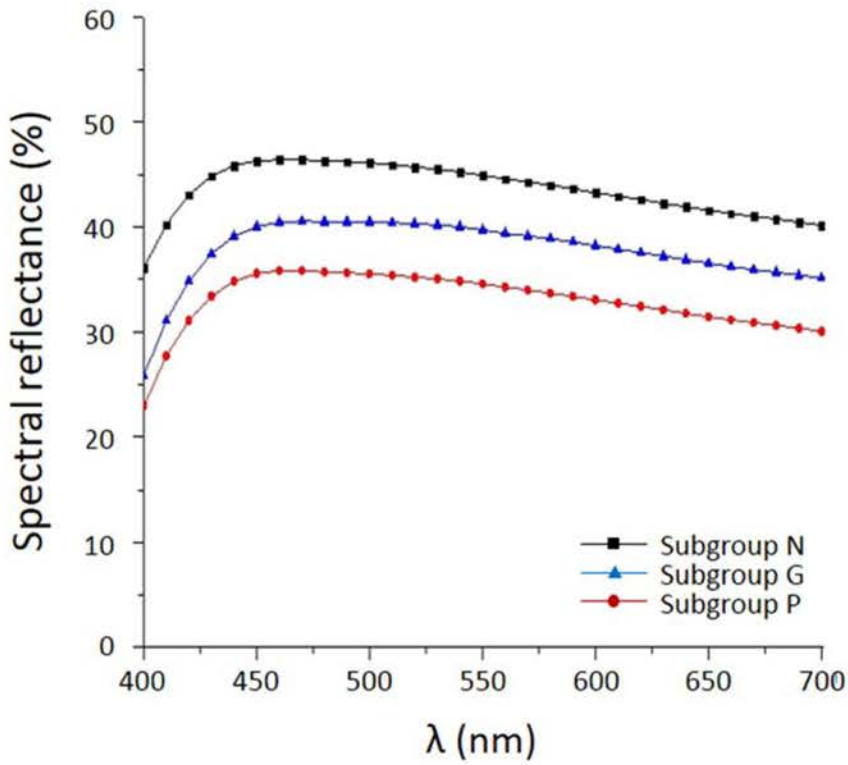

Fig. 21. Spectral reflectance of each subgroup in Group II against white background (Experiment II).

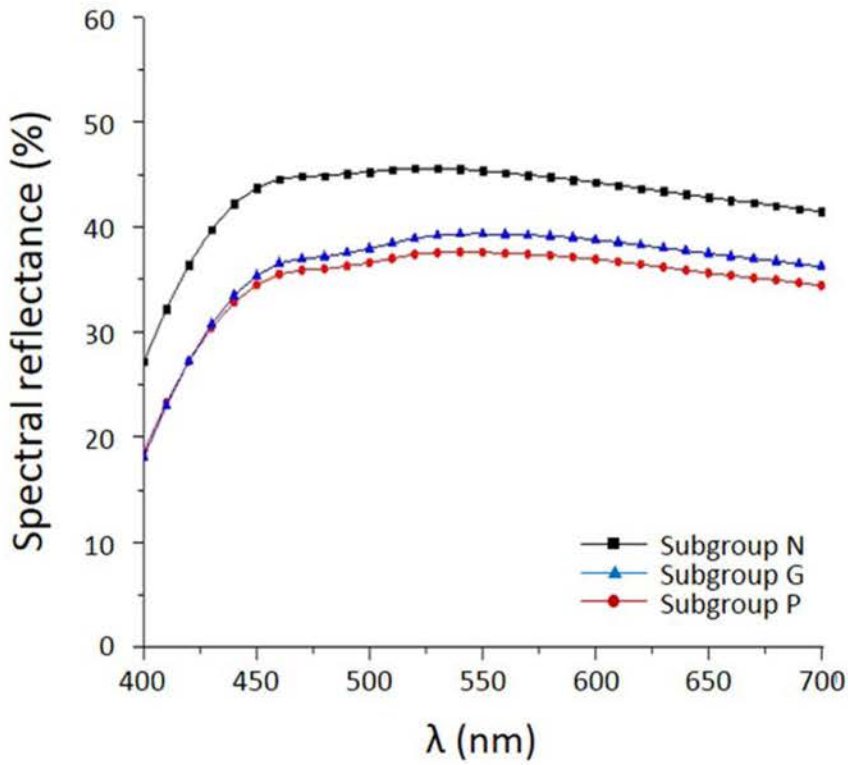

Fig. 22. Spectral reflectance of each subgroup in Group III against white background (Experiment II).

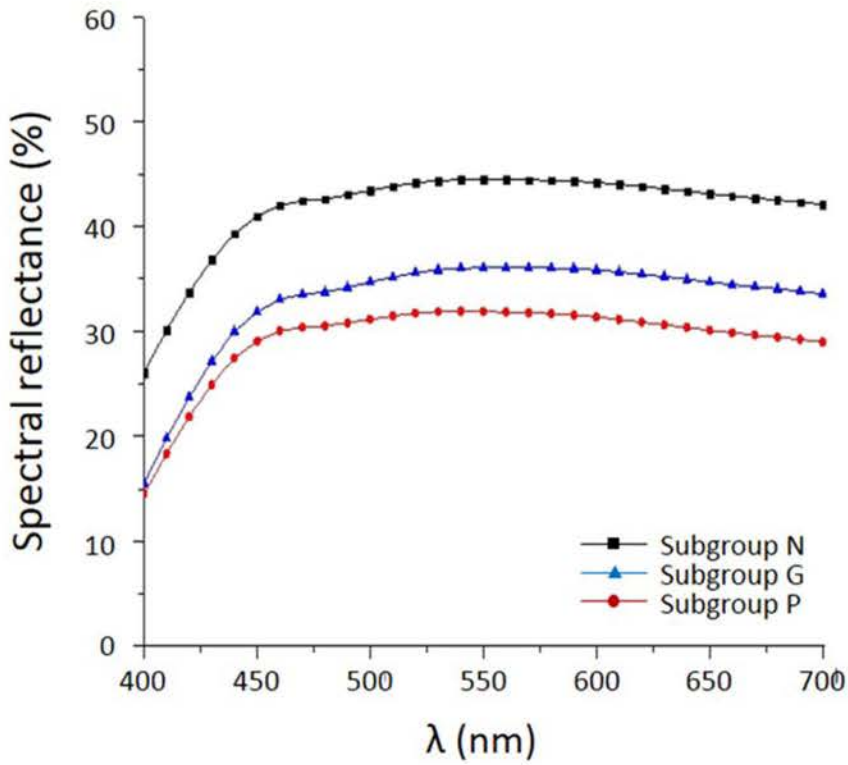

Fig. 23. Spectral reflectance of each subgroup in Group IV against white background (Experiment II).

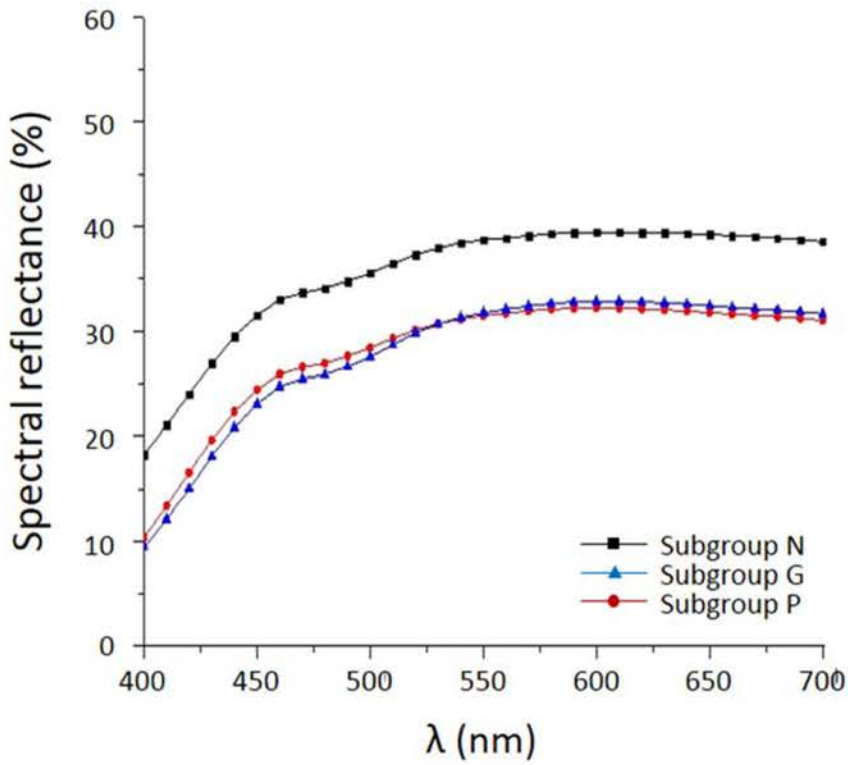

Fig. 24. Spectral reflectance of each subgroup in Group V against white background (Experiment II).

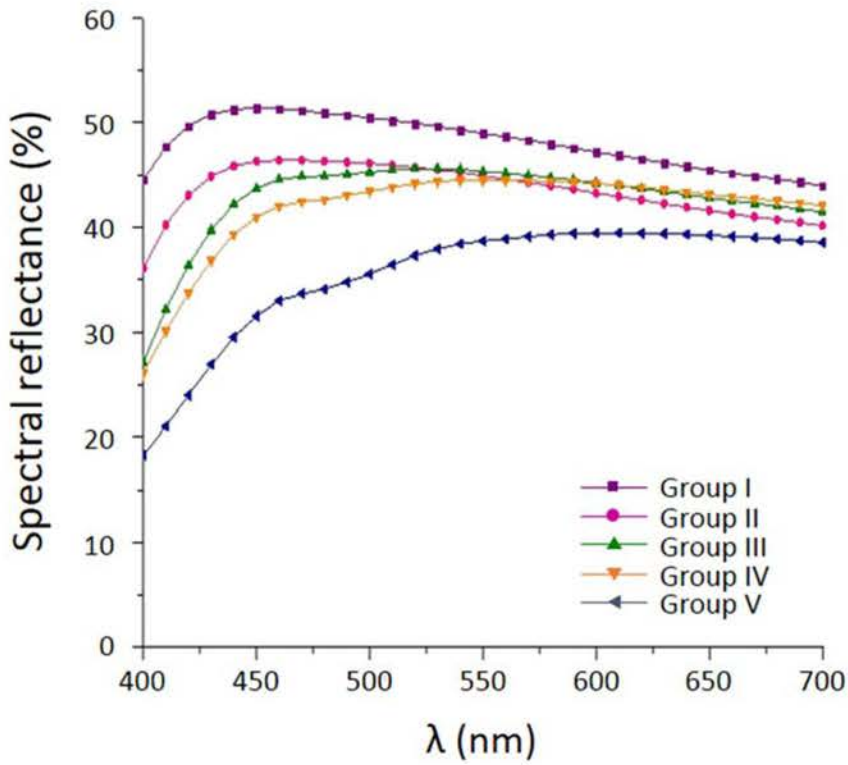

Fig. 25. Spectral reflectance of each group in Subgroup N (Experiment II).

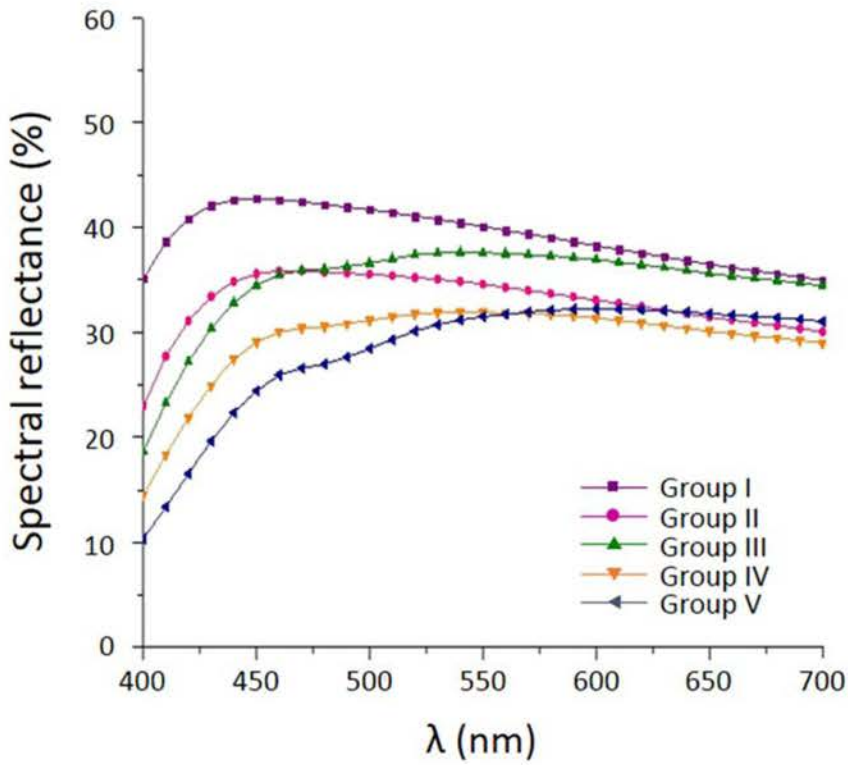

Fig. 26. Spectral reflectance of each group in Subgroup P (Experiment II).

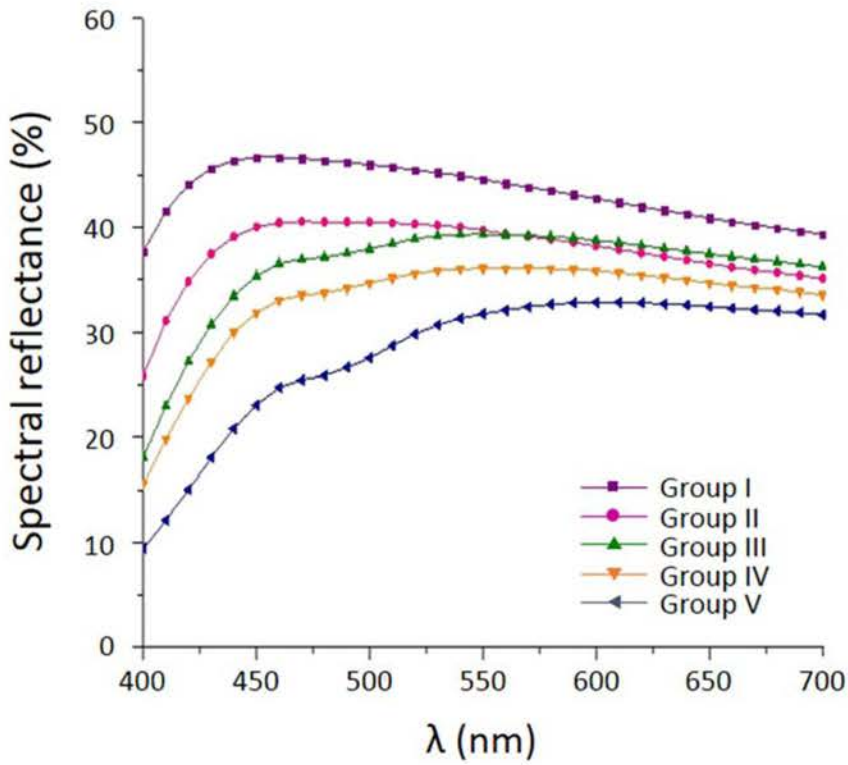

Fig. 27. Spectral reflectance of each group in Subgroup G (Experiment II).

applications increased in Subgroup N, P and G especially for the short wavelengths.

Color differences (ΔE^*_{ab}) between each pair of surface treatments within groups are shown in Table 10. Color difference between Subgroup N and P showed the highest values in comparison with the other pairs of surface treatments for the entire group ranged from 5.13 to 9.79 ΔE^*_{ab} units, which are clinically perceptible ($\Delta E^*_{ab} > 3.7$). Color difference between Subgroup N and G was in the range from 2.91 to 6.72 ΔE^*_{ab} units. A perceptible color difference was obtained between Subgroup N and G in Group III, IV and V. Color differences between Subgroup P and G are within the range of perceptibility threshold except Group II. Color differences between each group set in Subgroup N, P and G were shown in Table 11. Color difference between each pair of groups was in the range from 1.85 to 13.04 in Subgroup N, from 4.53 to 14.84 in Subgroup P, from 2.48 to 17.55 in Subgroup G, respectively. In general, a perceptible color difference was obtained in each group set.

Correlations between the number of coloring liquid applications and CIE L^*, a^* or b^* values in each surface treatment were identified. In all subgroups, CIE L^* tended to be decreased and CIE b^* value tended to be increased as the number of coloring liquid applications increased. There was a significant

Table 10. Color differences between each group set

Group	Subgroup set	ΔE^*_{ab}
I	N-P	5.98
	N-G	2.91
	P-G	3.17
II	N-P	7.17
	N-G	3.69
	P-G	3.85
III	N-P	5.19
	N-G	4.61
	P-G	1.38
IV	N-P	9.79
	N-G	6.72
	P-G	3.56
V	N-P	5.13
	N-G	5.93
	P-G	2.35

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

Table 11. Color differences between each group set

Subgroup	Group set	ΔE^*_{ab}
N	I-II	3.21
	I-III	5.61
	I-IV	7.12
	I-V	13.94
	II-III	3.89
	II-IV	5.68
	II-V	11.66
	III-IV	1.85
	III-V	8.45
	IV-V	7.14
P	I-II	4.70
	I-III	7.35
	I-IV	9.44
	I-V	14.84
	II-III	5.80
	II-IV	5.51
	II-V	11.69
	III-IV	4.53
	III-V	7.85
	IV-V	6.40
G	I-II	4.19
	I-III	8.03
	I-IV	9.87
	I-V	17.55
	II-III	5.01
	II-IV	6.18
	II-V	14.00
	III-IV	2.48
	III-V	9.64
	IV-V	7.85

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

correlation between the number of coloring liquid applications and CIE b^* value indicating r value to be 0.960 and R^2 to be 0.922 in Subgroup N, r value to be 0.948 and R^2 to be 0.899 in Subgroup P, and r value to be 0.962 and R^2 to be 0.925 in Subgroup G, respectively (Fig. 28). There was a negative correlation between the number of coloring liquid applications and CIE L^* value in each surface treatment (Fig. 29), whereas no significant correlation was found between the number of coloring liquid applications and CIE a^* value (Fig. 30).

Means and standard deviations for TP values within each group as a function of surface treatment are listed in Table 12. The statistical analyses showed no significant difference in TP values between each subgroup in Group I, II and II. There was no significant difference in TP values between Subgroup P and G in Group IV and V (Fig. 31).

Fig. 32 to 39 show the spectral transmittance behavior within groups or subgroups. There was no distinct difference between each subgroup in all groups. Fig. 37 shows that there was a decrease in spectral transmittance value with the increase of number of coloring liquid applications in Subgroup N and this tendency corresponds to Subgroup P and G (Fig. 38 and 39).

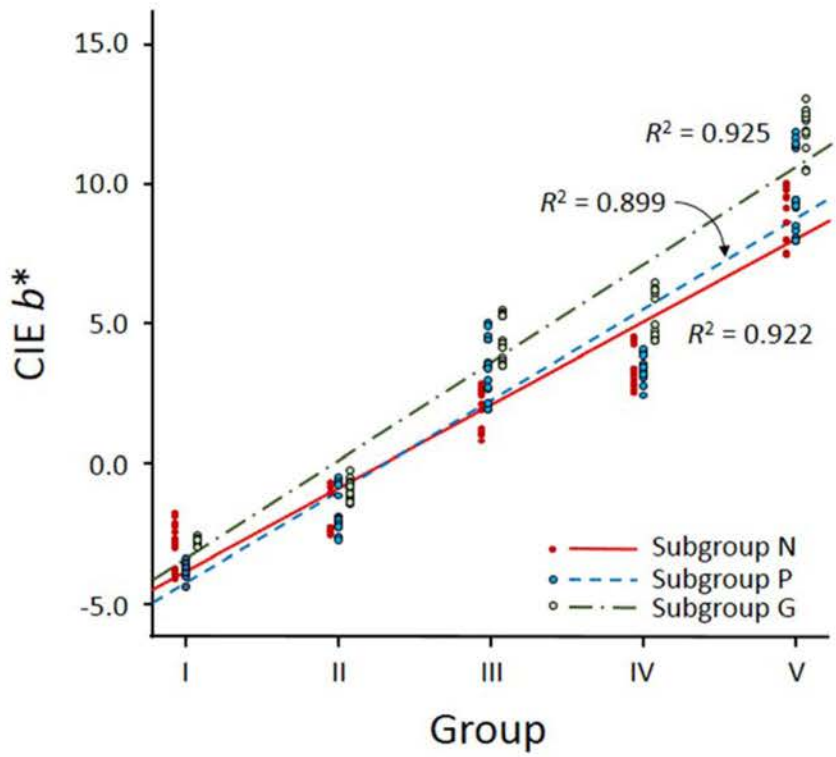

Fig. 28. Linear regression of CIE b^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

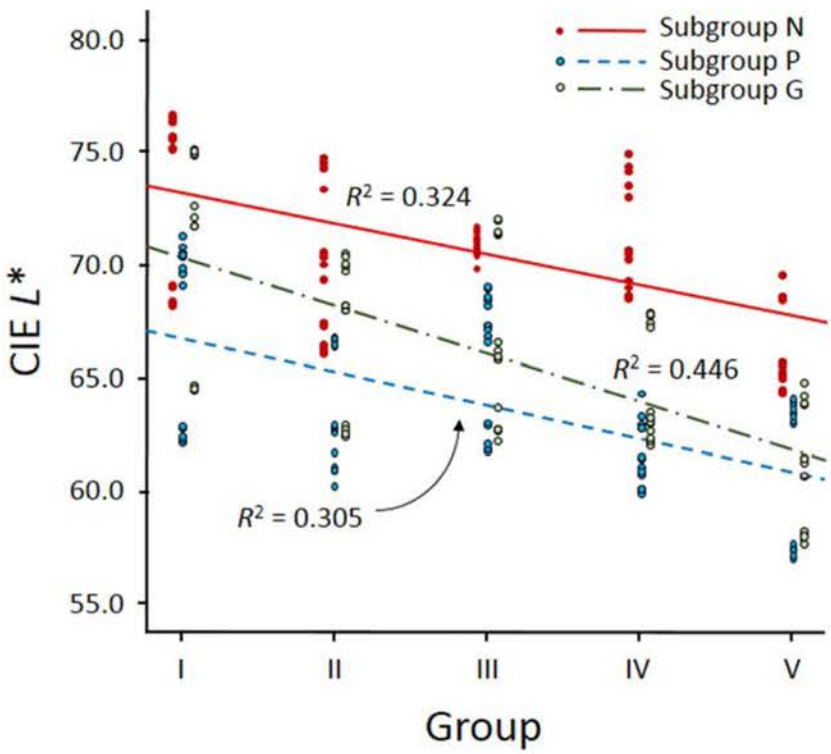

Fig. 29. Linear regression of CIE L^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

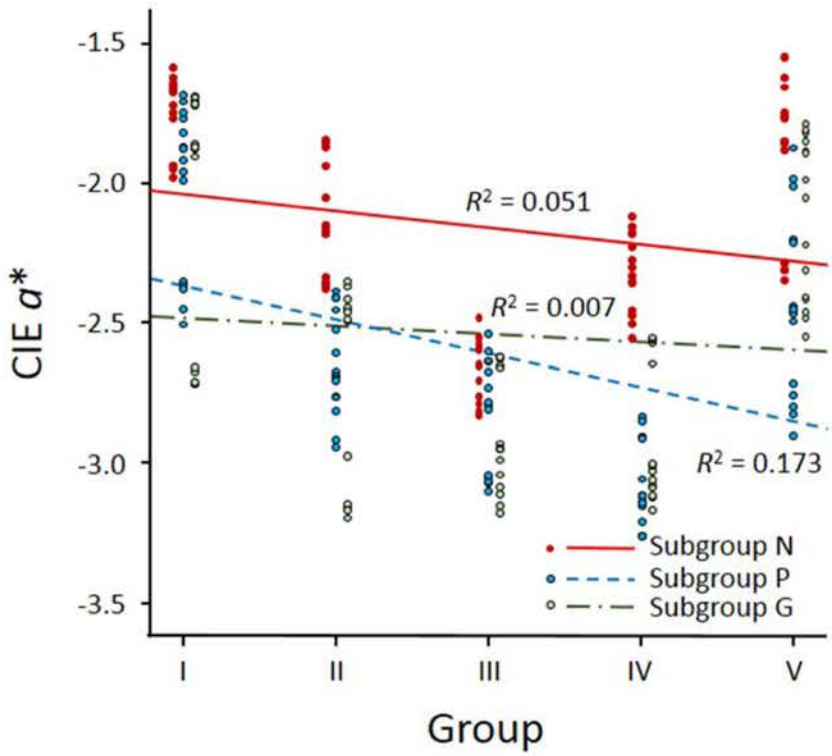

Fig. 30. Linear regression of CIE a^* values of each subgroup over a zero calibration box in the reflectance mode as a function of the number of coloring liquid applications (Experiment II).

Table 12. Means and standard deviations in parentheses for translucency parameter of each group as a function of surface treatment

	Surface treatment	Group				
		I	II	III	IV	V
TP	N	1.87[a] (0.48)	2.02[a] (0.41)	2.21[a] (0.34)	2.05[a] (0.42)	1.74[a] (0.41)
	P	1.96[a] (0.52)	1.85[a] (0.32)	2.13[a] (0.17)	2.19[a] (0.24)	1.85[a,b] (0.31)
	G	1.85[a] (0.24)	2.00[a] (0.46)	2.16[a] (0.41)	2.18[a] (0.28)	2.06[b] (0.17)

• Means with the same superscript letter in each group column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

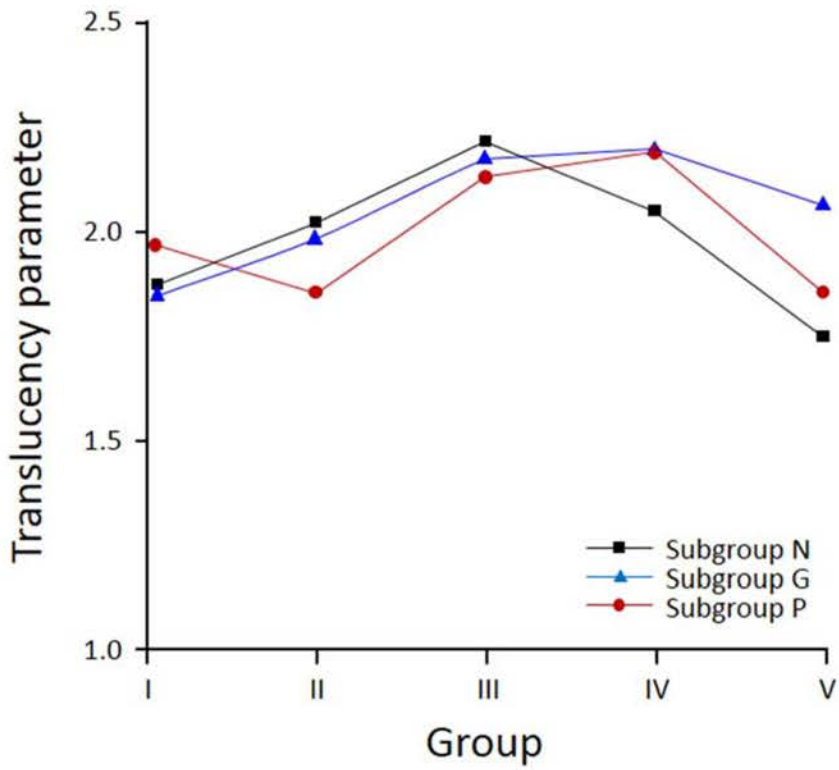

Fig. 31. Means of translucency parameter values of each subgroup as a function of the number of coloring liquid applications (Experiment II).

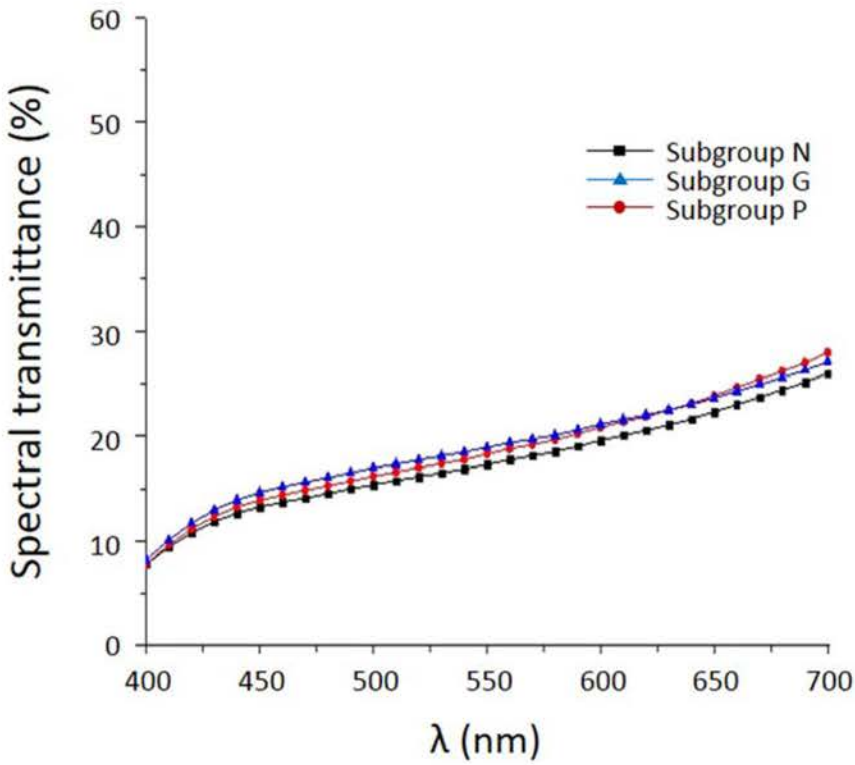

Fig. 32. Spectral transmittance of each subgroup in Group I (Experiment II).

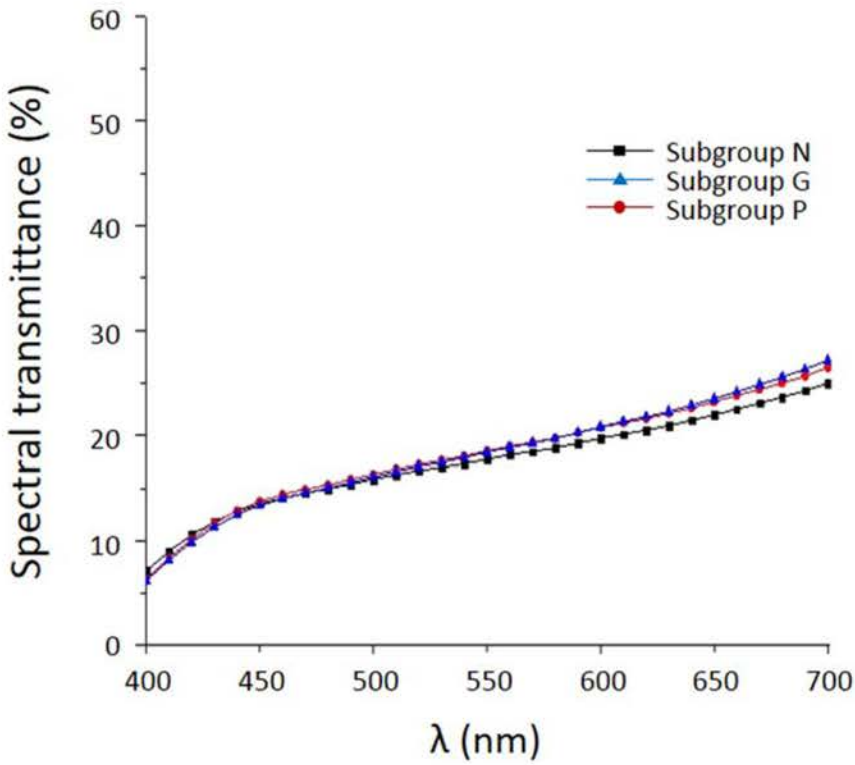

Fig. 33. Spectral transmittance of each subgroup in Group II (Experiment II).

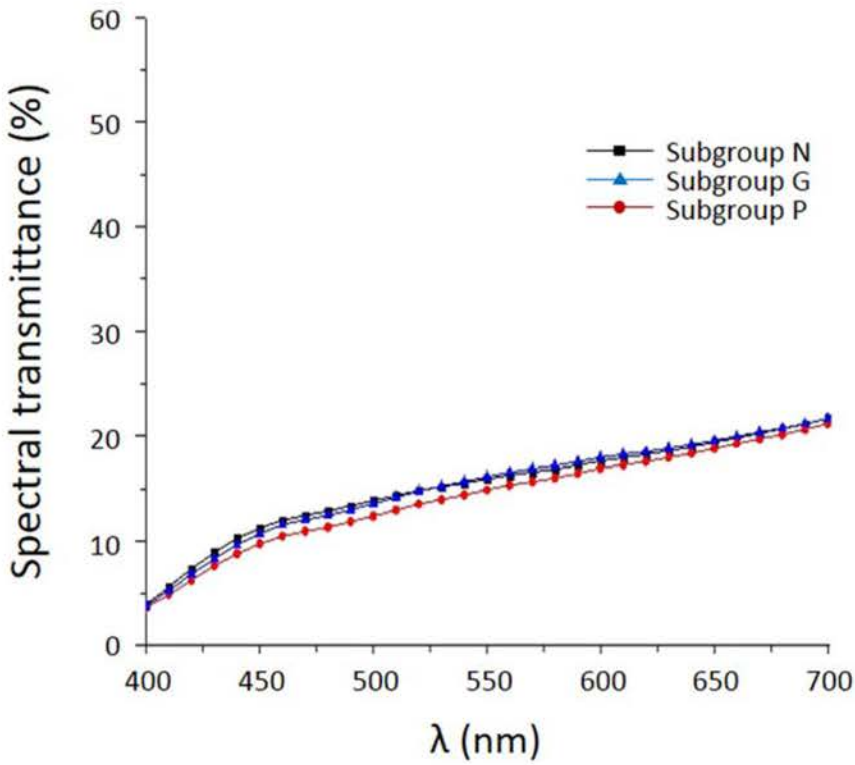

Fig. 34. Spectral transmittance of each subgroup in Group III (Experiment II).

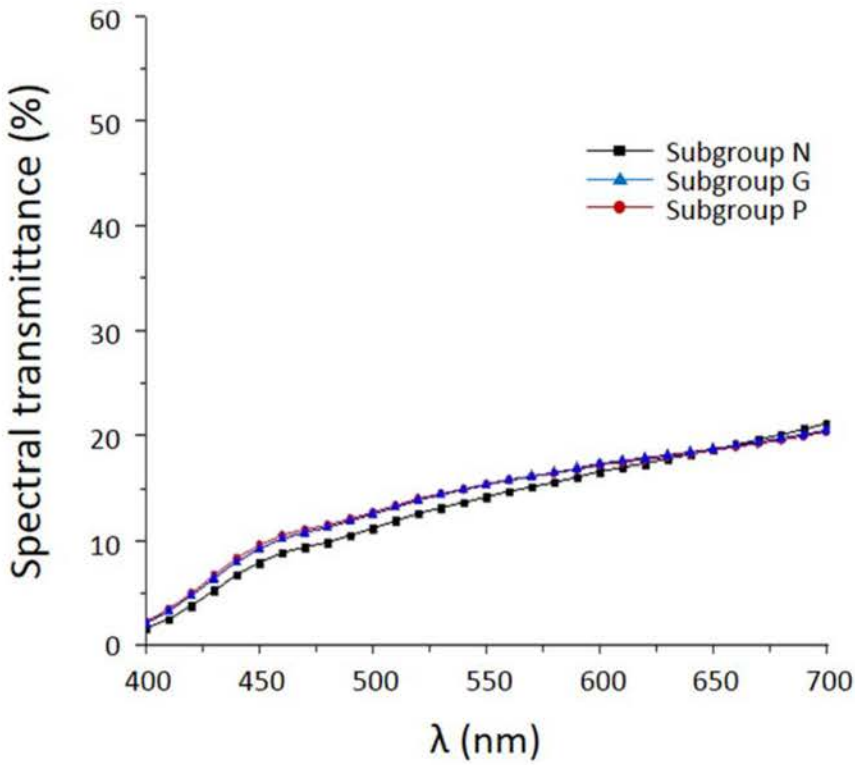

Fig. 35. Spectral transmittance of each subgroup in Group IV (Experiment II).

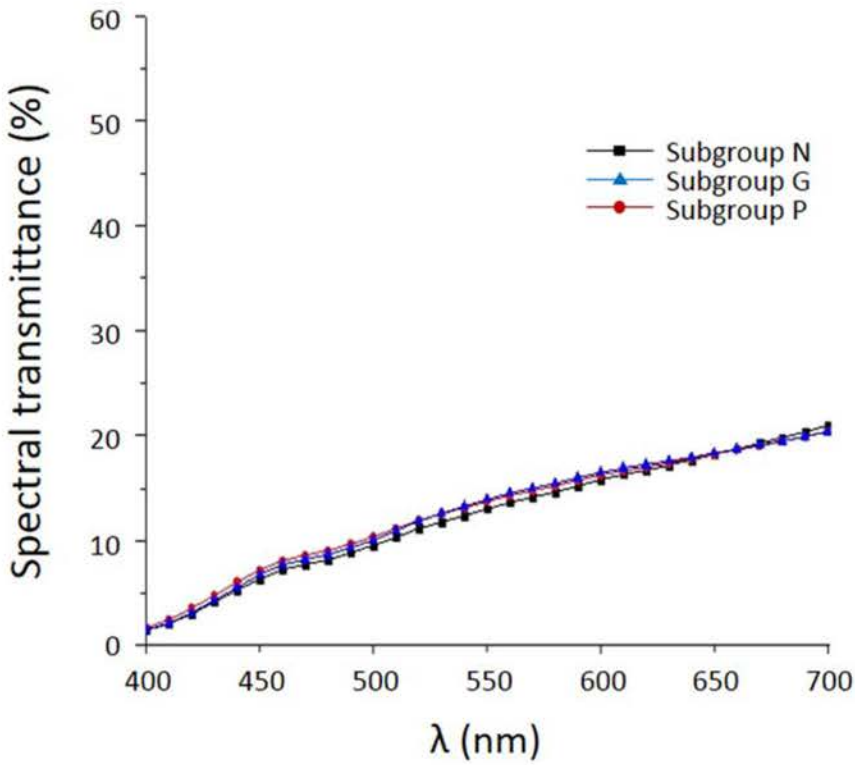

Fig. 36. Spectral transmittance of each subgroup in Group V (Experiment II).

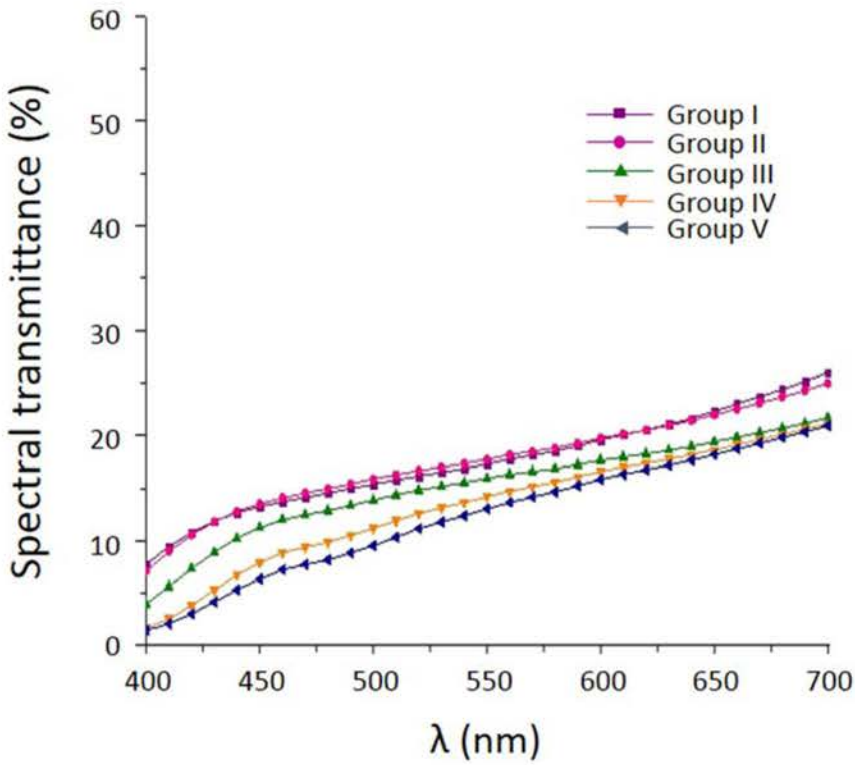

Fig. 37. Spectral transmittance of each group in Subgroup N (Experiment II).

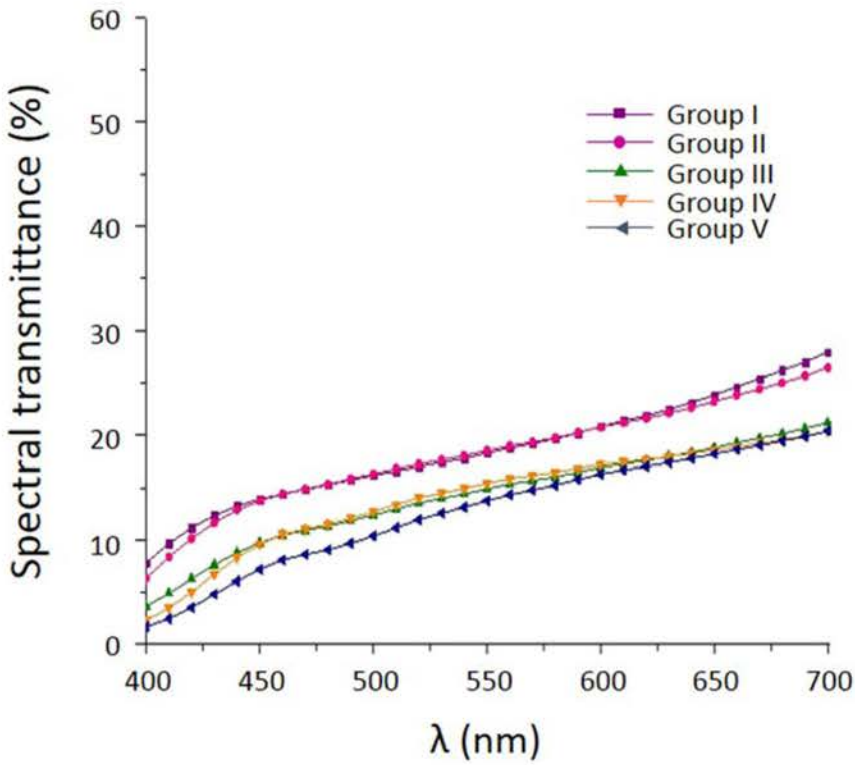

Fig. 38. Spectral transmittance of each group in Subgroup P (Experiment II).

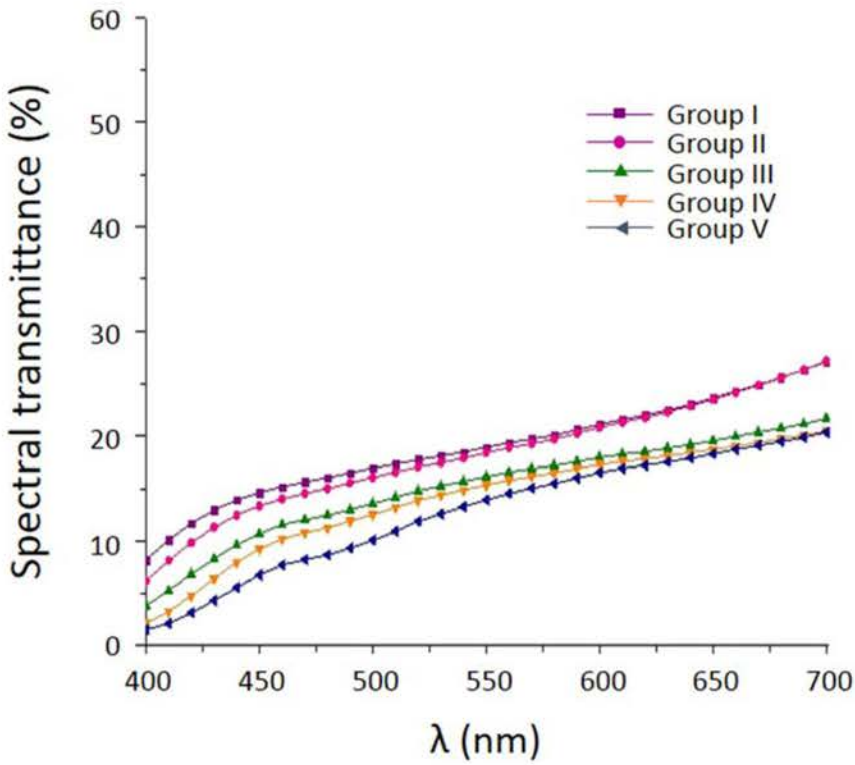

Fig. 39. Spectral transmittance of each group in Subgroup G (Experiment II).

3.3. Experiment III. Effect of the amount of thickness reduction on the color and translucency of monolithic zirconia

Means and standard deviations of L^*, a^* and b^* values over a zero calibrating box in the reflectance mode within each group as a function of the amount of thickness reduction are listed in Table 13-1 to 13-5. In Group I (Table 13-1), there was a significant difference between Subgroup 0 and other subgroups for L^*, and b^* value. In Group II (Table 13-2), there was a significant difference among Subgroup 0, Subgroup 1 and other subgroups for L^* value, and b^* value. In Group III (Table 13-3), there was a significant difference between Subgroup 0 and other subgroups for L^* value and there was a significant difference among Subgroup 0, Subgroup 1 and other subgroups for b^* value. In Group IV (Table 13-4), there was a significant difference between Subgroup 0 and other subgroups for L^* value, and b^* value generally decreased. In Group V (Table 13-5), there was a significant difference between Subgroup 0 and other subgroups for L^* value, and b^* value generally decreased. On the contrary, a^* values generally increased as the amount of thickness reduction increased in all groups.

Correlation between L^*, a^* or b^* value and the amount of thickness reduction is presented in Fig. 40 to 42. There were negative, but weak correlations between L^* value and the amount of thickness reduction in Group I, II and III (Fig. 40). There were positive correlations between a^* value and the amount

Table 13-1. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group I as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
I	0	75.20 (1.96)	$-1.74^{a,b}$ (0.13)	-2.93 (0.51)
	1	68.19^{b} (0.79)	-1.78^{a} (0.04)	-4.86^{a} (0.26)
	2	68.41^{b} (0.82)	$-1.60^{c,d,e}$ (0.05)	$-4.57^{a,b}$ (0.11)
	3	$67.72^{a,b}$ (0.43)	$-1.55^{d,e,f}$ (0.07)	-4.74^{a} (0.12)
	4	66.65^{a} (0.28)	$-1.62^{c,d}$ (0.03)	-4.86^{a} (0.15)
	5	$67.10^{a,b}$ (0.40)	$-1.67^{b,c}$ (0.04)	-4.69^{a} (0.18)
	6	$67.24^{a,b}$ (0.55)	$-1.59^{d,e}$ (0.03)	$-4.60^{a,b}$ (0.14)
	7	66.44^{a} (0.29)	$-1.62^{c,d}$ (0.05)	$-4.59^{a,b}$ (0.08)
	8	$67.41^{a,b}$ (0.71)	$-1.51^{e,f}$ (0.07)	$-4.27^{b,c}$ (0.21)
	9	68.15^{b} (0.51)	$-1.46^{f,g}$ (0.05)	-4.13^{c} (0.14)
	10	67.76^{b} (1.06)	-1.40^{g} (0.08)	-4.08^{c} (0.24)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-2. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group II as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
II	0	75.35 (2.78)	-2.38 (0.19)	0.35 (0.20)
	1	73.25 (2.86)	-1.72^{a} (0.09)	-3.04 (0.82)
	2	68.11^{a} (0.15)	$-1.62^{a,b}$ (0.05)	$-4.34^{b,c}$ (0.05)
	3	66.99^{a} (0.52)	$-1.62^{a,b}$ (0.06)	-4.85^{a} (0.17)
	4	67.02^{a} (0.15)	$-1.61^{a,b}$ (0.03)	$-4.69^{a,b}$ (0.08)
	5	67.33^{a} (0.26)	$-1.64^{a,b}$ (0.03)	$-4.54^{a,b,c}$ (0.04)
	6	67.67^{a} (0.54)	$-1.60^{a,b}$ (0.04)	$-4.50^{a,b,c}$ (0.21)
	7	67.42^{a} (0.25)	$-1.59^{a,b}$ (0.04)	$-4.33^{b,c}$ (0.05)
	8	67.20^{a} (0.33)	$-1.55^{b,c}$ (0.03)	$-4.38^{b,c}$ (0.11)
	9	68.01^{a} (0.13)	-1.44^{c} (0.60)	-4.22^{c} (0.07)
	10	67.87^{a} (0.19)	-1.29 (0.16)	-4.16^{c} (0.06)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-3. Means and standard deviations in parentheses for CIE *L**, *a** and *b** values over a zero calibration box in the reflectance mode within Group III as a function of the amount of thickness reduction

Group	Subgroup	*L**	*a**	*b**
III	0	72.67 (1.88)	-2.72 (0.10)	3.34 (0.88)
	1	67.51[b] (1.10)	-2.32 (0.17)	-2.04 (0.71)
	2	67.22[a,b] (0.45)	-1.83 (0.07)	-3.80[c] (0.11)
	3	67.33[a,b] (0.35)	-1.61[a,b] (0.05)	-4.62[a.b] (0.16)
	4	66.28[a] (0.34)	-1.63[a] (0.03)	-4.86[a] (0.13)
	5	67.45[a,b] (0.28)	-1.66[a] (0.02)	-4.52[a,b] (0.06)
	6	67.71[b] (0.75)	-1.55[a,b] (0.04)	-4.46[a,b] (0.19)
	7	67.03[a,b] (0.45)	-1.61[a] (0.04)	-4.40[a,b] (0.10)
	8	67.62[b] (0.20)	-1.50[b,c] (0.04)	-4.25[b,c] (0.09)
	9	68.23[b] (0.35)	-1.41[c,d] (0.06)	-4.20[b,c] (0.09)
	10	67.97[b] (0.22)	-1.37[d] (0.04)	-4.13[b,c] (0.11)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-4. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group IV as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
IV	0	72.70 (1.19)	-2.21^{b} (0.11)	5.79^{a} (0.33)
	1	64.38^{a} (1.44)	-2.52^{a} (0.14)	4.85^{a} (0.50)
	2	$65.42^{a,b}$ (0.55)	-2.62^{a} (0.10)	$1.45^{b,c}$ (1.65)
	3	65.69^{b} (0.79)	-2.56^{a} (0.12)	1.60^{b} (1.94)
	4	66.04^{b} (1.18)	-2.20^{b} (0.35)	-0.76^{c} (3.62)
	5	67.43^{c} (0.40)	-1.89^{c} (0.24)	-3.34^{d} (1.34)
	6	67.55^{c} (0.26)	-1.62^{d} (0.03)	-4.43^{d} (0.10)
	7	67.43^{c} (0.62)	$-1.66^{c,d}$ (0.12)	-4.15^{d} (0.44)
	8	67.61^{c} (0.25)	-1.55^{d} (0.04)	-4.25^{d} (0.10)
	9	68.22^{c} (0.12)	-1.46^{d} (0.04)	-4.13^{d} (0.12)
	10	67.89^{c} (0.29)	-1.43^{d} (0.04)	-4.09^{d} (0.10)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

Table 13-5. Means and standard deviations in parentheses for CIE L^*, a^* and b^* values over a zero calibration box in the reflectance mode within Group V as a function of the amount of thickness reduction

Group	Subgroup	L^*	a^*	b^*
V	0	68.97 (2.91)	$-1.85^{b,c}$ (0.56)	9.01 (2.36)
	1	64.29^{a} (1.39)	-2.65^{a} (0.05)	4.66 (0.52)
	2	$65.47^{a,b}$ (0.59)	-2.60^{a} (0.05)	3.01 (1.04)
	3	$66.91^{b,c}$ (0.89)	-2.10^{b} (0.15)	-2.40 (0.35)
	4	67.33^{c} (0.59)	$-1.77^{c,d}$ (0.07)	-3.81^{a} (0.14)
	5	67.40^{c} (0.28)	$-1.70^{c,d,e}$ (0.05)	-4.26^{a} (0.21)
	6	67.73^{c} (0.18)	$-1.60^{c,d,e,f}$ (0.04)	-4.34^{a} (0.06)
	7	$66.97^{b,c}$ (0.16)	$-1.58^{c,d,e,f}$ (0.05)	-4.51^{a} (0.06)
	8	67.72^{c} (0.14)	$-1.50^{d,e,f}$ (0.07)	-4.36^{a} (0.07)
	9	68.90 (0.70)	-1.41^{f} (0.06)	-4.00^{a} (0.22)
	10	67.76^{c} (0.54)	$-1.42^{e,f}$ (0.05)	-4.06^{a} (0.15)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

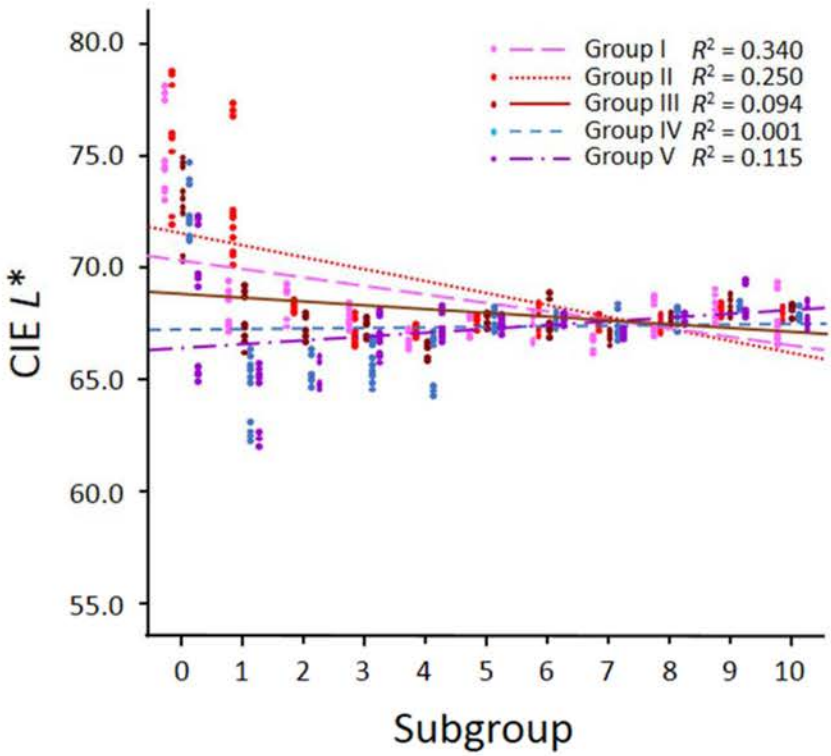

Fig. 40. Linear regression of CIE L^* values of each group as a function of the amount of thickness reduction (Experiment III).

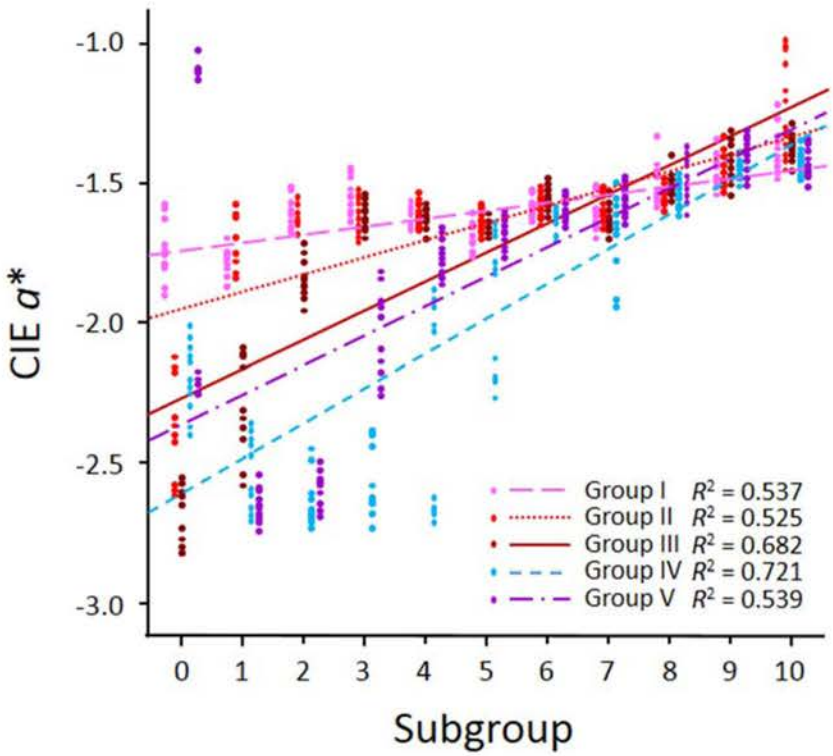

Fig. 41. Linear regression of CIE a^* values of each group as a function of the amount of thickness reduction (Experiment III).

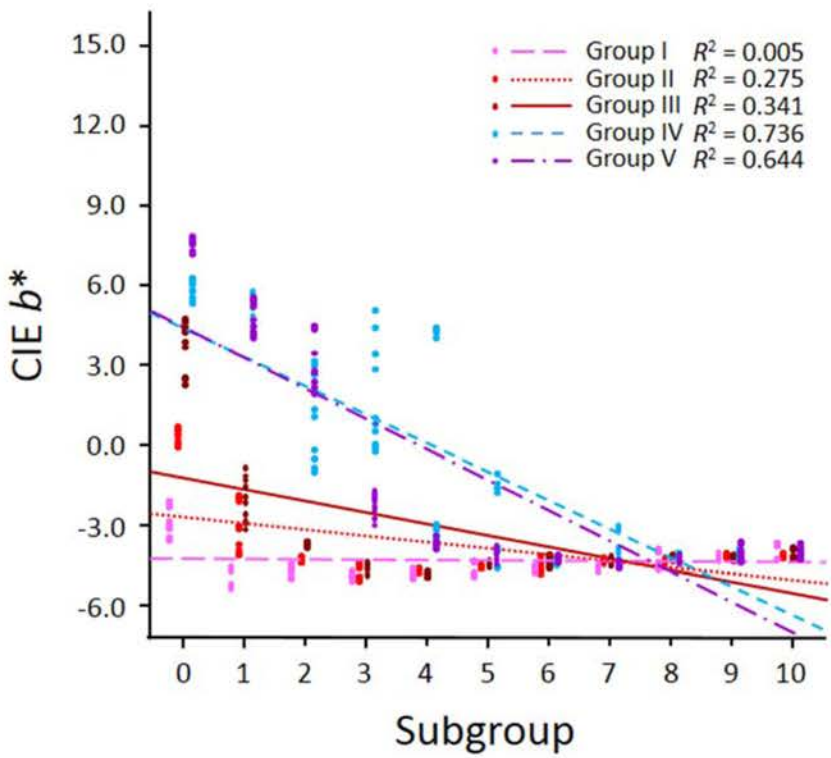

Fig. 42. Linear regression of CIE b^* values of each group as a function of the amount of thickness reduction (Experiment III).

of thickness reduction in all groups ($0.72 < r < 0.85$, $0.52 < R^2 < 0.73$, Fig. 41). There were negative correlations between b^* value and the amount of thickness reduction in all groups ($-0.86 < r < -0.07$, $0.00 < R^2 < 0.74$, Fig. 42).

Fig. 43 to 45 represented means of L^*, a^* or b^* for each group as a function of the amount of thickness reduction. Significant decrease in L^* values after initial 0.1 mm reduction was observed in all groups (Fig. 43). For a^* and b^* values, there was no significant difference between groups from *circa* 0.6 mm reduction (Fig. 44 and 45).

Average reflectance was calculated at each wavelength in the spectral range of 400 to 700 nm with an interval of 10 nm. Fig. 46 to 50 show spectral reflectance curves of average reflectance of specimens against the white background within groups. In Group I (Fig. 46), there was a significant difference between Subgroup 0 and other subgroups through the entire spectrum in the range of 400 to 700 nm and the values of spectral reflectance in other subgroups were lower than those in Subgroup 0. In Group II to IV (Fig. 47, 48 and 49), reflectance of Subgroup 0 was lower than that of other subgroups in the short wavelength range and higher in the long wavelength range. The crossing point occurred at longer wavelength with increasing the

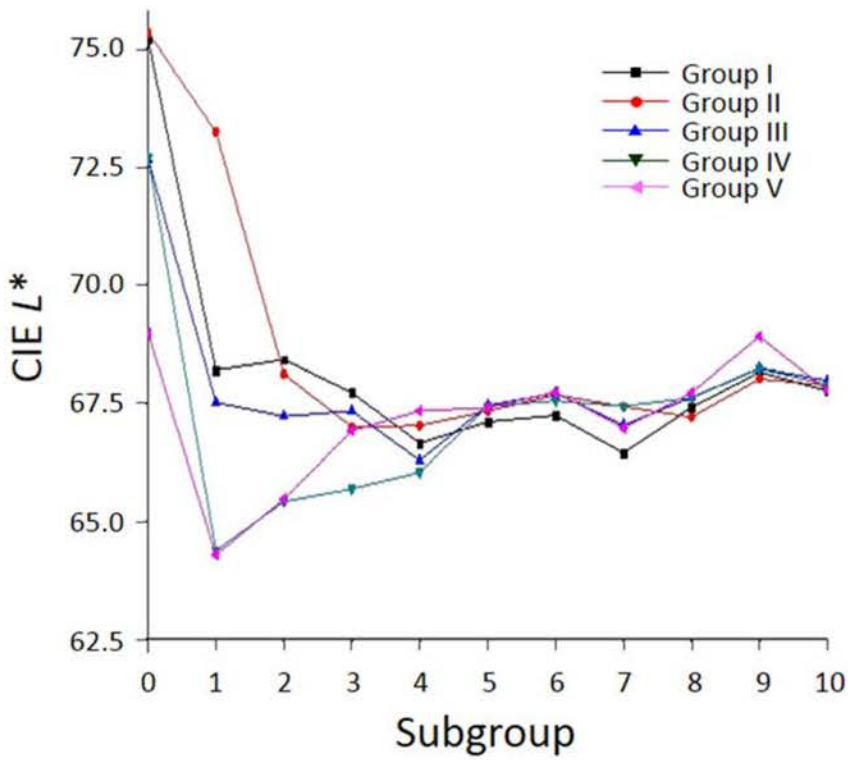

Fig. 43. Means of CIE L^* values of each group as a function of the amount of thickness reduction (Experiment III).

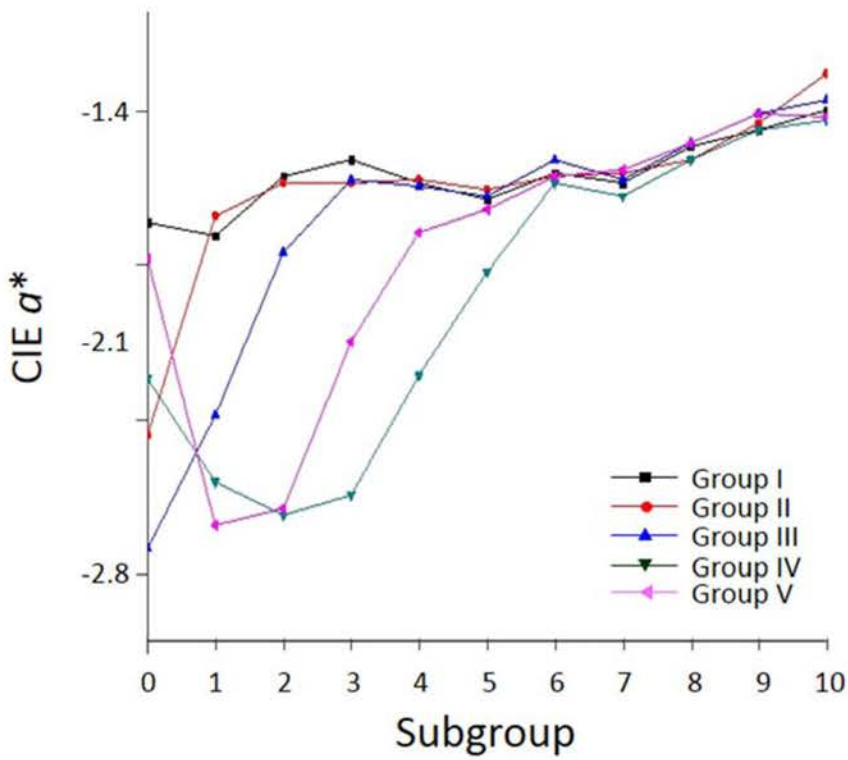

Fig. 44. Means of CIE *a** values of each group as a function of the amount of thickness reduction (Experiment III).

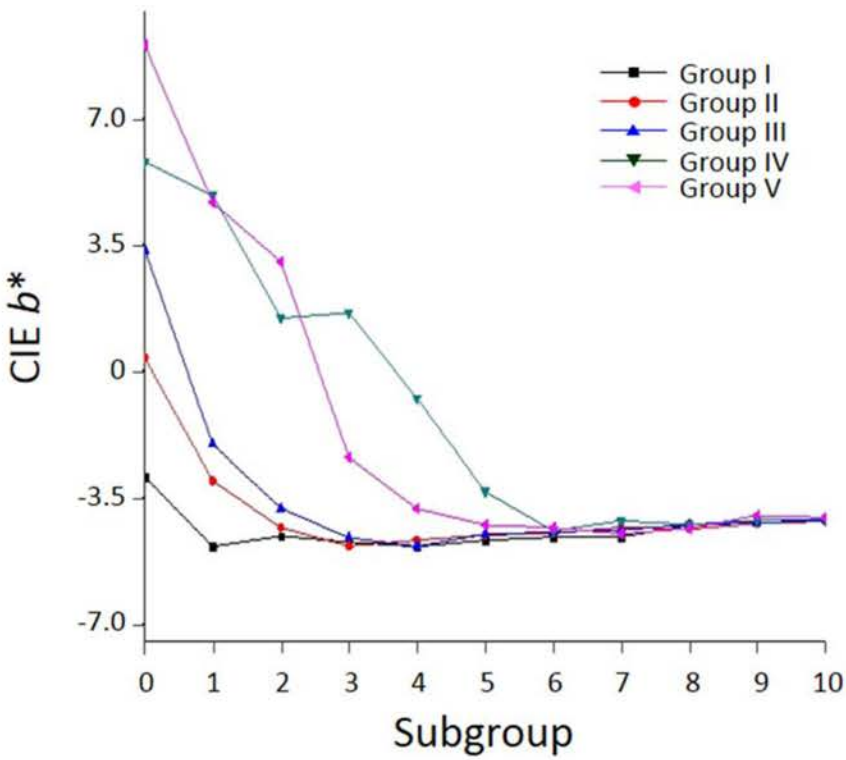

Fig. 45. Means of CIE *b** values of each group as a function of the amount of thickness reduction (Experiment III).

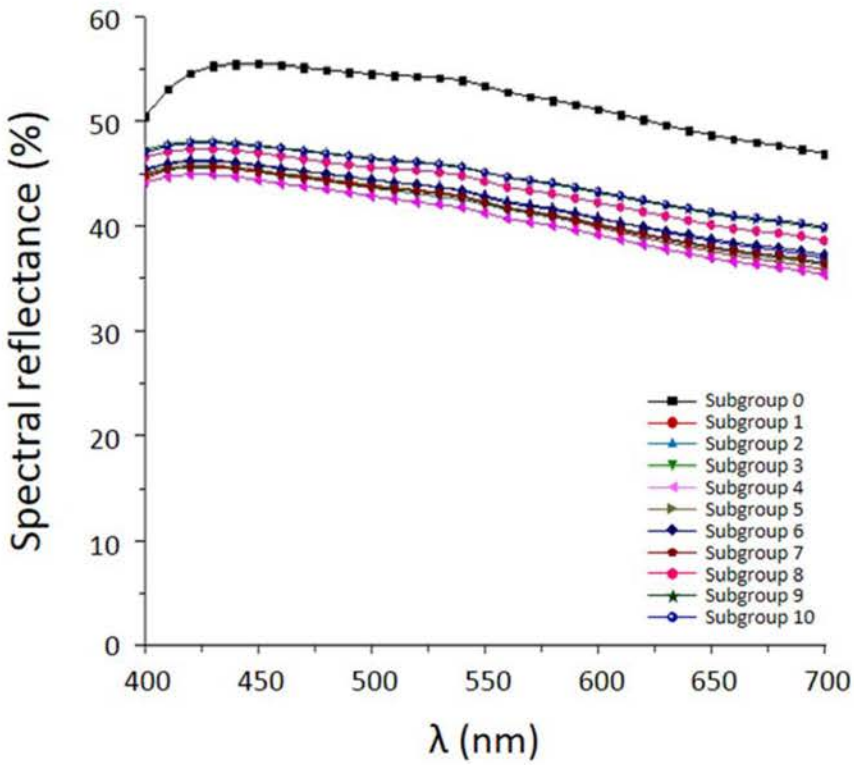

Fig. 46. Spectral reflectance of each subgroup in Group I (Experiment III).

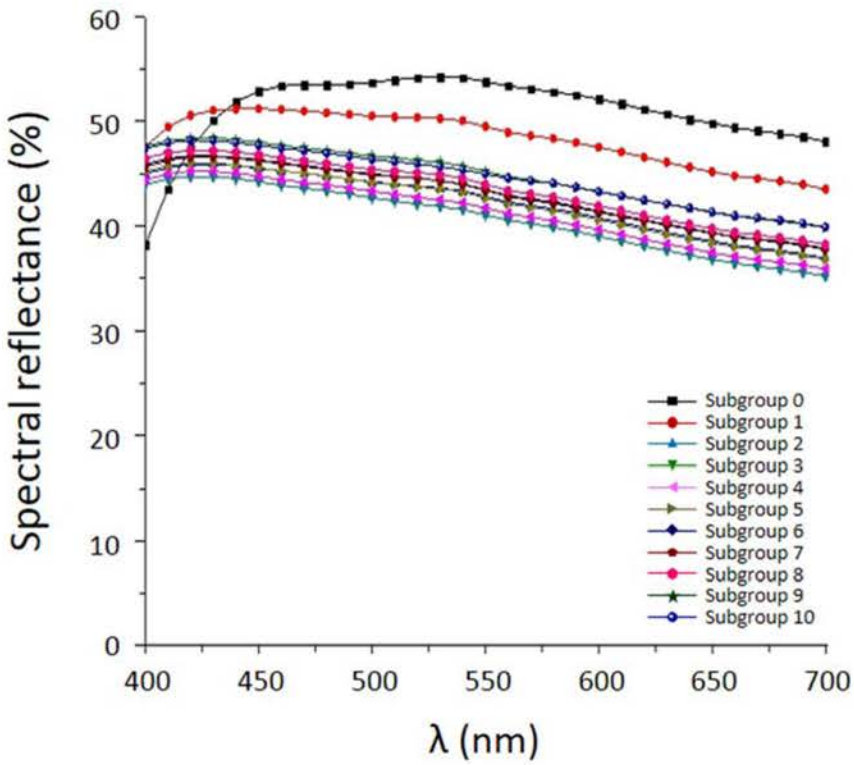

Fig. 47. Spectral reflectance of each subgroup in Group II (Experiment III).

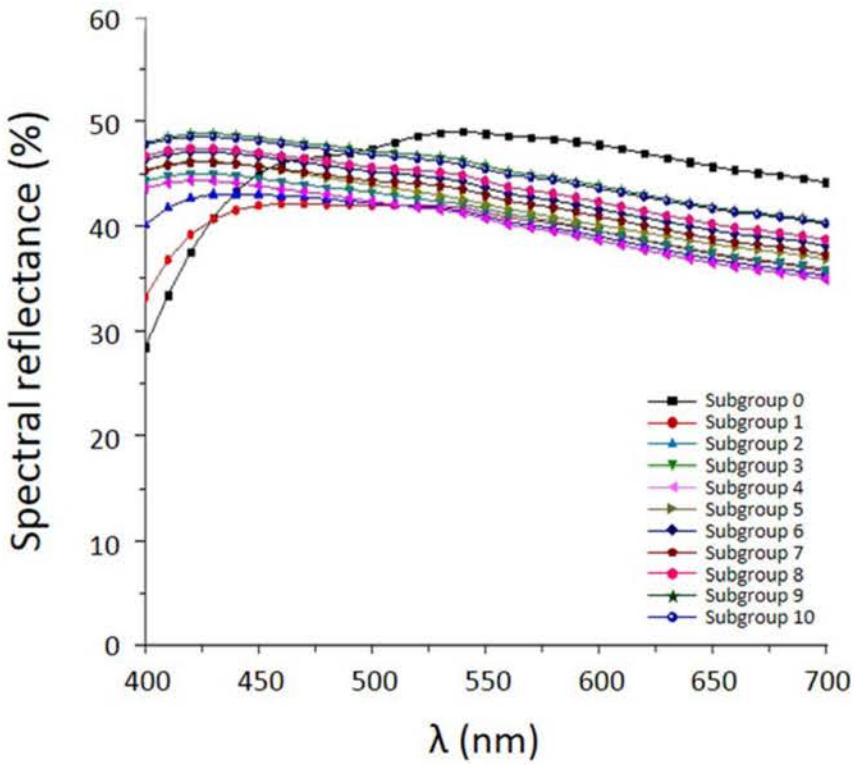

Fig. 48. Spectral reflectance of each subgroup in Group III (Experiment III).

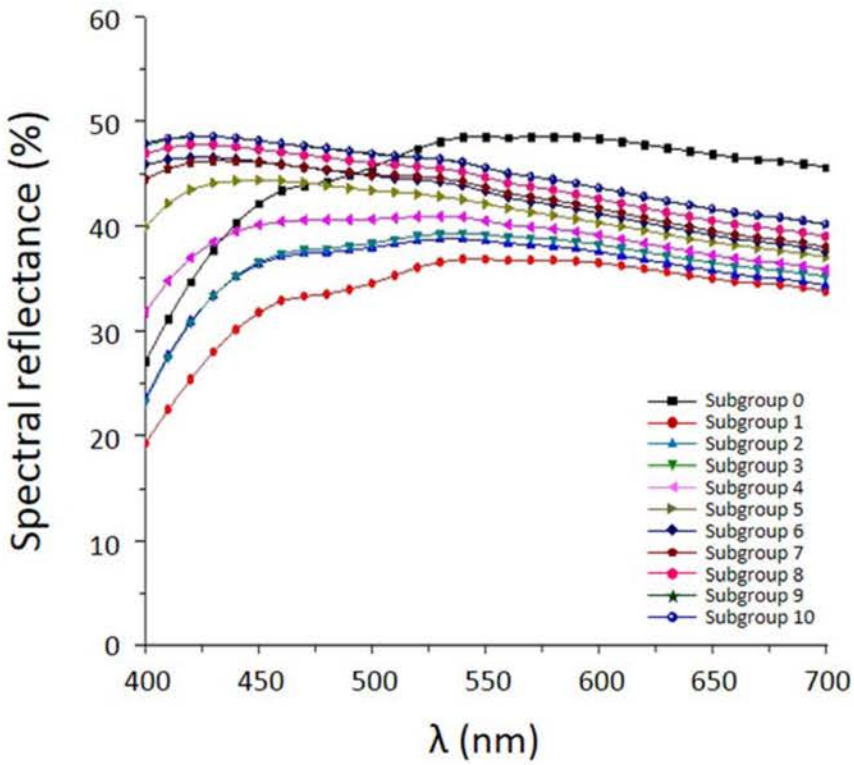

Fig. 49. Spectral reflectance of each subgroup in Group IV (Experiment III).

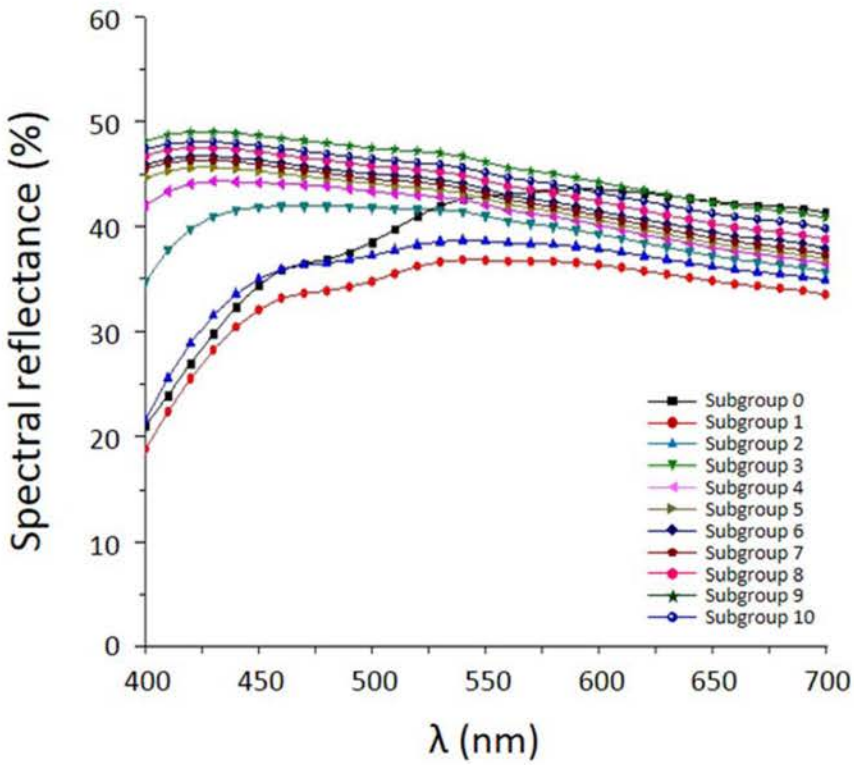

Fig. 50. Spectral reflectance of each subgroup in Group V (Experiment III).

number of coloring liquid applications. However, this trend was not so clear in Group V (Fig. 50).

Color differences (ΔE^*_{ab}) between each subgroup set are shown in Table 14. Color differences between Subgroup 0 and Subgroup 1 ranged from 4.04 to 8.38 ΔE^*_{ab} units, which were clinically perceptible ($\Delta E^*_{ab} > 3.7$). Fig. 51 shows ΔE^*_{ab} units for each group calculated by the means of L^*, a^* and b^* values between Subgroup 0 and each subgroup. Color differences between Subgroup 1 and 2 were within the range of perceptibility threshold ($\Delta E^*_{ab} < 3.7$) except Group II. Color differences between Subgroup 2 and 3 were within the range of perceptibility threshold except Group V. Color differences between Subgroup 3 and 4, 4 and 5, 5 and 6, 6 and 7, 7 and 8, 8 and 9, 9 and 10 were within the range of perceptibility threshold in all groups.

Means and standard deviations of TP for each group are listed in Table 15. TP values generally increased as the amount of thickness reduction increased in all groups (Fig. 52). Highly significant correlations were found out between TP values and the amount of thickness reduction in all groups ($r > 0.94$, $R^2 > 0.89$, $P < 0.001$, Fig. 53). Average transmittance was calculated at each wavelength in the spectral range of 400 to 700 nm with an interval of 10 nm and spectral curves are shown in Fig. 54 to 58. Each subgroup exhibited similar spectral behavior through the entire spectrum in the range of 400 to

Table 14. Color differences (ΔE^*_{ab}) between each group set

Subgroup set	Group				
	I	II	III	IV	V
0-1	7.27	4.04	7.46	8.38	6.44
0-2	6.99	8.65	9.03	8.49	6.98
0-3	7.70	9.88	9.65	8.18	11.59
0-4	8.76	9.76	10.45	9.34	12.92
0-5	8.28	9.42	9.49	10.55	13.36
0-6	8.13	9.11	9.32	11.46	13.41
0-7	8.91	9.24	9.64	11.27	13.67
0-8	7.91	9.46	9.19	11.28	13.43
0-9	7.15	8.70	8.85	10.91	13.02
0-10	7.53	8.80	8.93	11.02	13.13
1-2	0.40	5.30	1.85	3.56	2.03
1-3	0.54	6.53	2.68	3.50	7.54
1-4	1.55	6.45	3.15	5.87	9.04
1-5	1.11	6.11	2.57	8.77	9.49
1-6	1.00	5.77	2.55	9.85	9.69
1-7	1.78	5.98	2.52	9.54	9.62
1-8	1.02	6.20	2.36	9.70	9.71
1-9	0.79	5.39	2.45	9.82	9.89
1-10	0.96	5.52	2.34	9.67	9.46
2-3	0.71	1.24	0.86	0.31	5.62
2-4	1.78	1.15	1.43	2.34	7.12
2-5	1.31	0.81	0.77	5.24	7.58
2-6	1.17	0.47	0.87	6.33	7.76
2-7	1.97	0.70	0.67	6.02	7.74
2-8	1.05	0.92	0.69	6.19	7.79
2-9	0.52	0.24	1.16	6.34	7.90
2-10	0.83	0.44	0.94	6.18	7.53

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

Table 14 (Continued). Color differences (ΔE^*_{ab}) between each group set

Subgroup set	Group				
	I	II	III	IV	V
3-4	1.08	0.17	1.08	2.42	1.51
3-5	0.63	0.47	0.17	5.28	1.97
3-6	0.50	0.77	0.41	6.38	2.17
3-7	1.29	0.68	0.37	6.08	2.18
3-8	0.56	0.53	0.48	6.24	2.21
3-9	0.75	1.22	1.01	6.36	2.66
3-10	0.67	1.16	0.84	6.21	1.99
4-5	0.49	0.34	1.22	2.94	0.46
4-6	0.65	0.67	1.49	4.00	0.69
4-7	0.34	0.53	0.88	3.70	0.81
4-8	0.97	0.36	1.48	3.87	0.73
4-9	1.68	1.10	2.07	4.07	1.63
4-10	1.37	1.04	1.86	3.88	0.61
5-6	0.18	0.35	0.29	1.13	0.35
5-7	0.67	0.23	0.44	0.84	0.52
5-8	0.54	0.22	0.36	0.98	0.38
5-9	1.21	0.77	0.87	1.19	1.55
5-10	0.93	0.74	0.71	0.99	0.49
6-7	0.80	0.30	0.69	0.31	0.77
6-8	0.38	0.49	0.24	0.21	0.10
6-9	1.04	0.47	0.60	0.76	1.24
6-10	0.76	0.49	0.46	0.52	0.34
7-8	1.02	0.23	0.62	0.23	0.76
7-9	1.78	0.62	1.23	0.82	2.00
7-10	1.43	0.57	1.01	0.52	0.92
8-9	0.76	0.83	0.61	0.63	1.24
8-10	0.41	0.74	0.39	0.34	0.32
9-10	0.40	0.21	0.27	0.34	1.14

• ΔE^*_{ab} denotes CIE 1976 a,b (CIELAB) color difference.

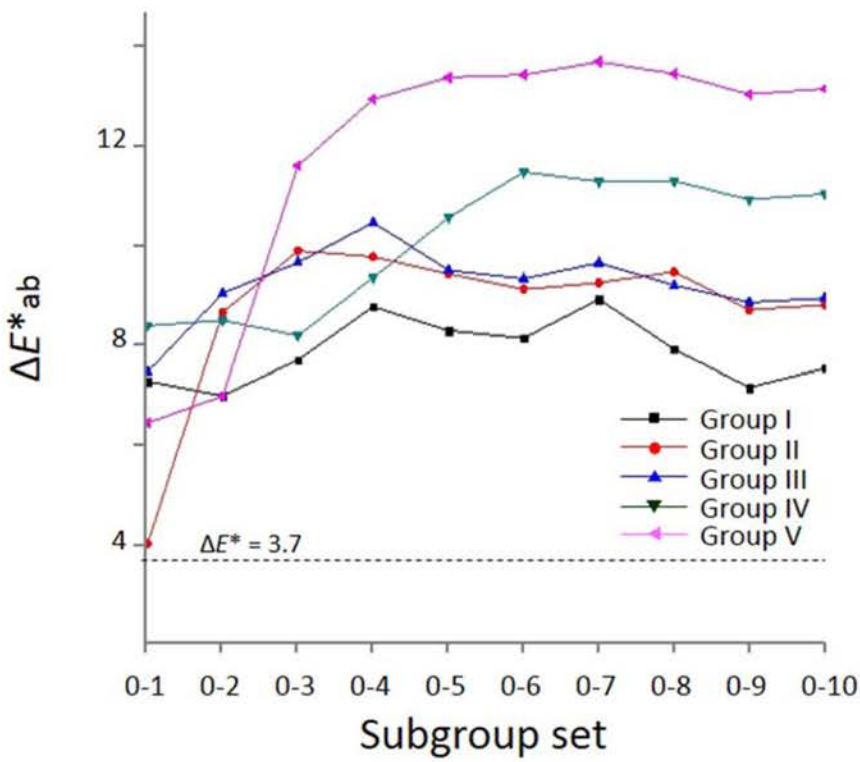

Fig. 51. ΔE^*_{ab} units between Subgroup 0 and each subgroup for each group (Experiment III).

Table 15. Means and standard deviations in parentheses for translucency parameter of each group as a function of the amount of thickness reduction

Subgroup	Group				
	I	II	III	IV	V
0	2.76[a] (0.39)	2.72[a] (0.31)	2.43 (0.10)	2.27 (0.19)	2.29 (0.13)
1	2.84[a] (0.13)	2.48[a] (0.29)	2.93[a] (0.22)	2.93[a] (0.18)	2.90[a] (0.21)
2	3.12[a,b] (0.20)	3.34[b,c] (0.09)	2.99[a] (0.13)	3.10[a,b] (0.14)	3.07[a] (0.08)
3	3.24[b] (0.06)	3.27[b] (0.12)	3.31[a,b] (0.08)	3.49[b,c] (0.22)	3.30[a,b] (0.24)
4	3.71[c] (0.10)	3.65[c,d] (0.09)	3.71[b] (0.12)	3.81[c,d] (0.17)	3.60[b,c] (0.12)
5	4.01[c,d] (0.08)	4.00[d,e] (0.13)	3.44[b] (0.35)	3.48[b,c] (0.23)	3.89[c,d] (0.11)
6	4.19[d,e] (0.09)	4.18[e,f] (0.11)	4.28[c] (0.16)	4.20[d,e] (0.08)	4.20[d] (0.09)
7	4.57[e,f] (0.18)	4.44[f] (0.25)	4.48[c,d] (0.22)	4.60[e] (0.22)	4.64[e] (0.22)
8	4.97[g,h] (0.16)	4.98[g] (0.18)	4.77[d,e] (0.27)	5.02[f] (0.27)	4.77[e] (0.32)
9	4.82[f,g] (0.18)	5.03[g] (0.33)	5.18[e,f] (0.41)	5.04[f] (0.35)	4.73[e] (0.50)
10	5.21[h] (0.58)	5.04[g] (0.41)	5.20[f] (0.48)	5.34[f] (0.52)	5.19 (0.51)

• Means with the same superscript letter in each column are not significantly different from each other based on multiple comparison Scheffé test ($P > 0.05$).

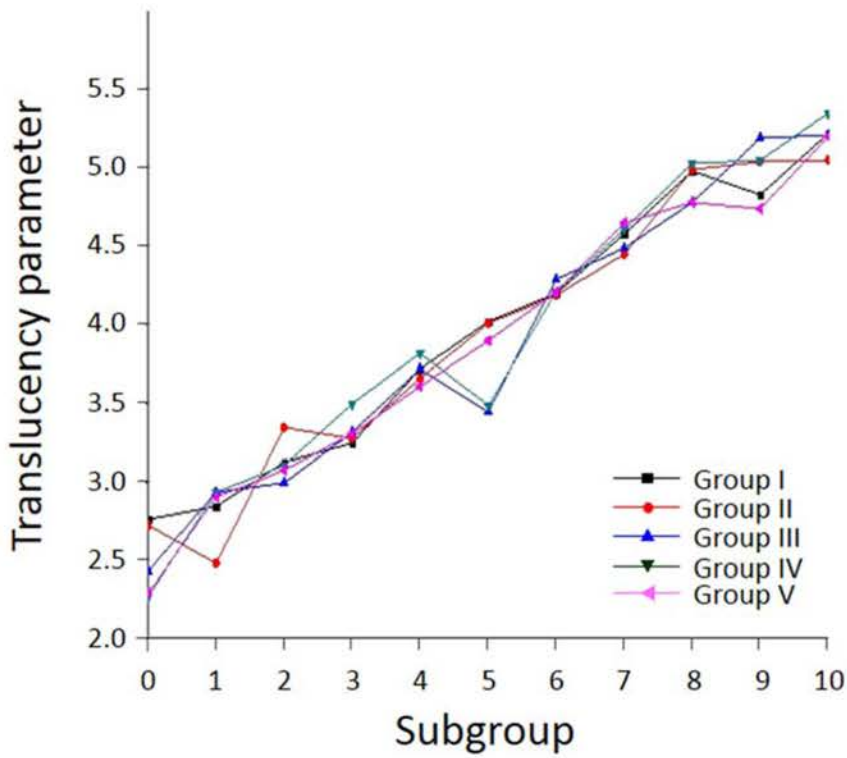

Fig. 52. Means of translucency parameter values for each group as a function of the amount of thickness reduction (Experiment III).

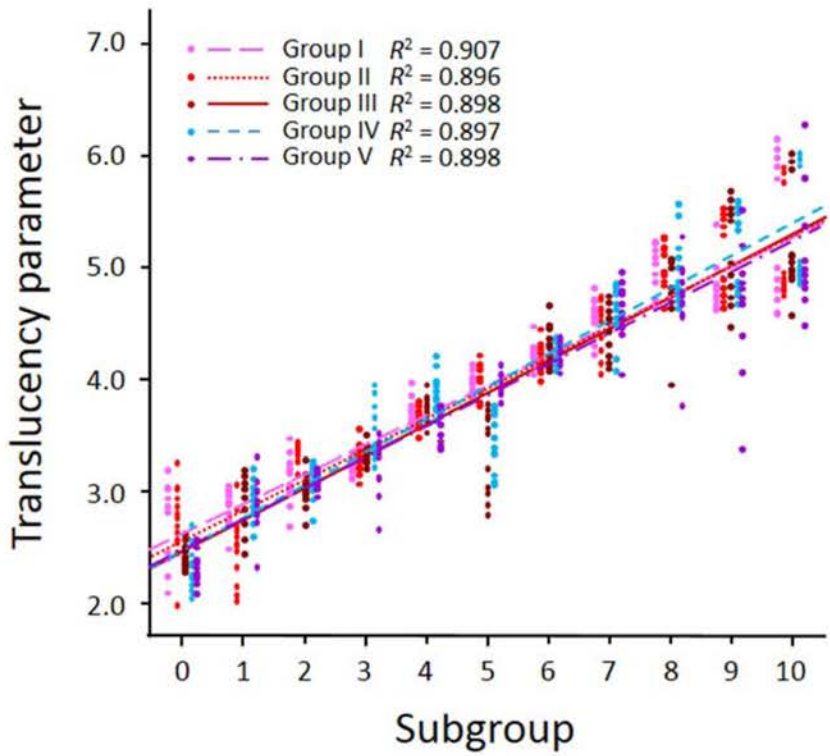

Fig. 53. Linear regression of translucency parameter values of each group as a function of the amount of thickness reduction (Experiment III).

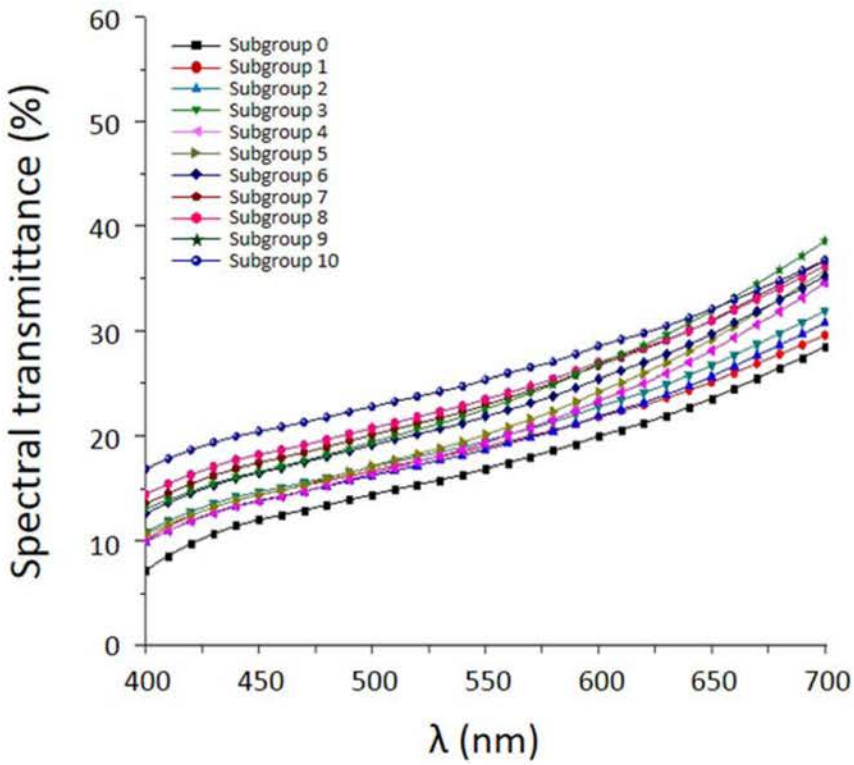

Fig. 54. Spectral reflectance of each subgroup in Group I (Experiment III).

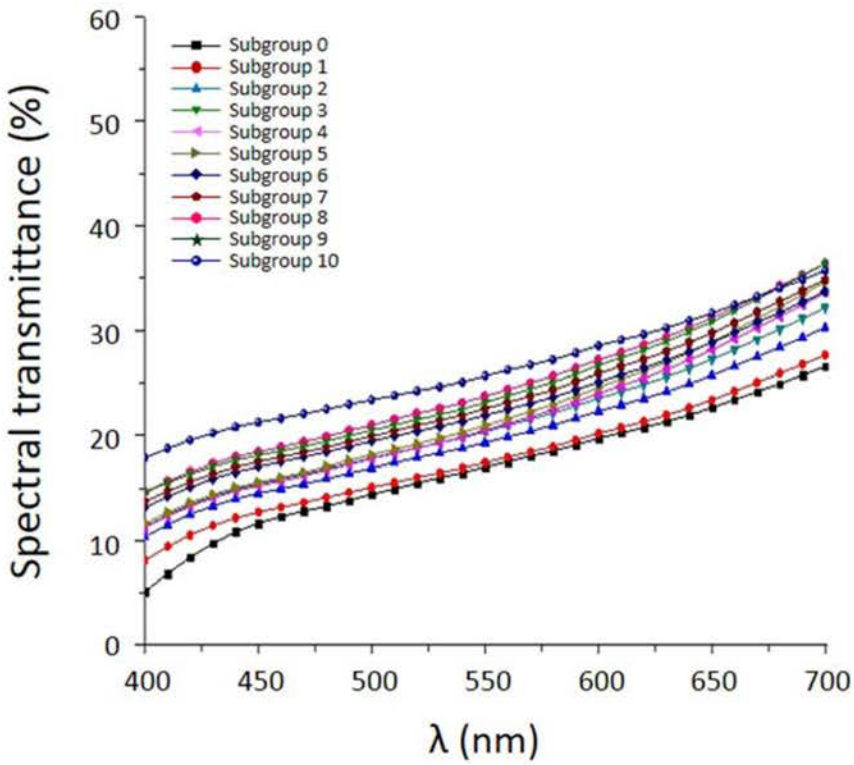

Fig. 55. Spectral reflectance of each subgroup in Group II (Experiment III).

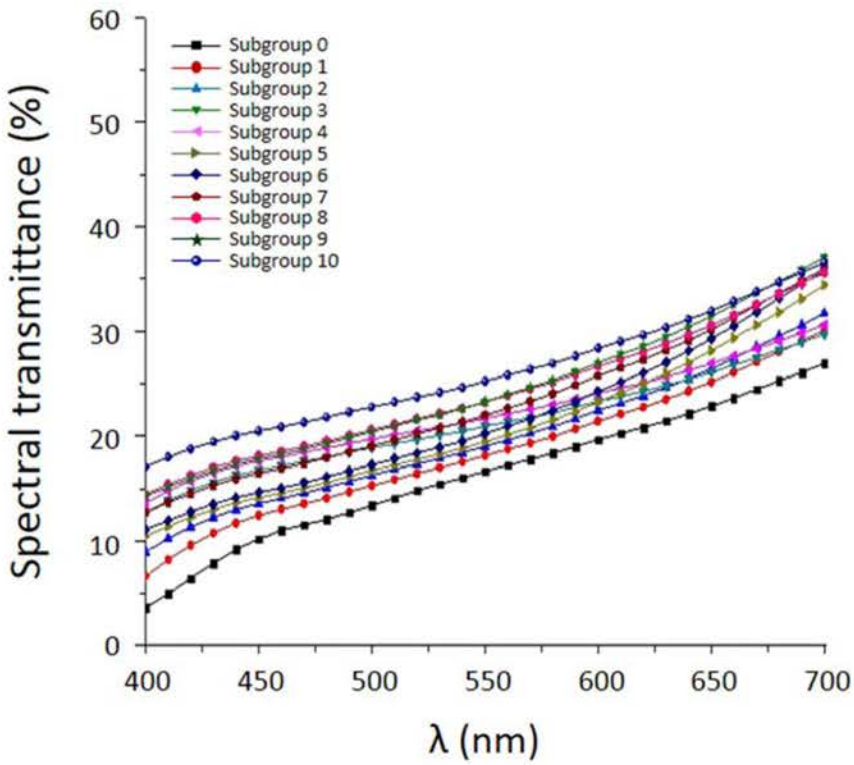

Fig. 56. Spectral reflectance of each subgroup in Group III (Experiment III).

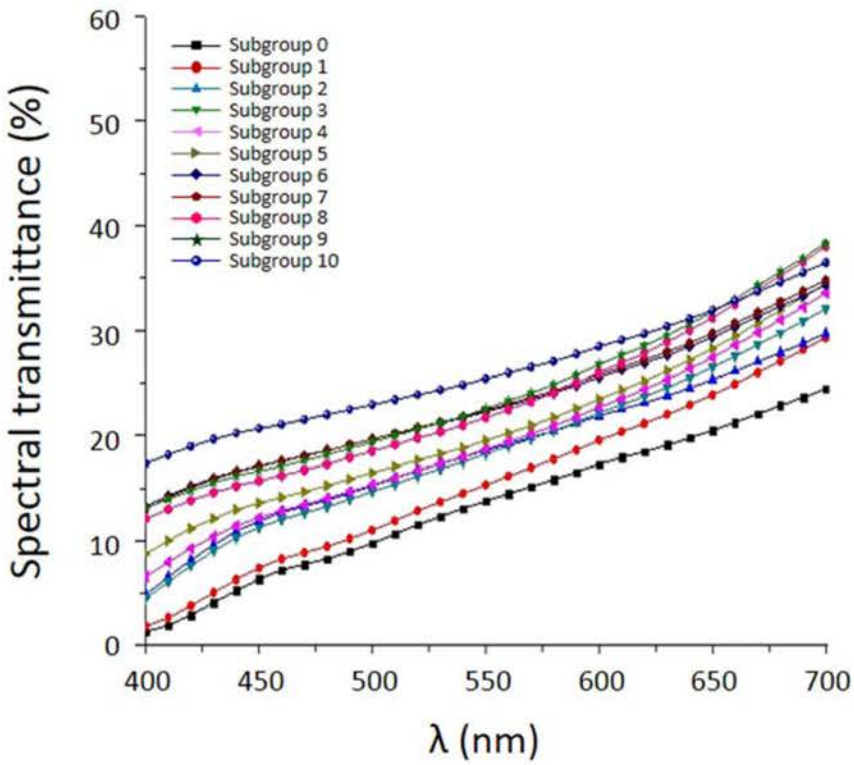

Fig. 57. Spectral reflectance of each subgroup in Group IV (Experiment III).

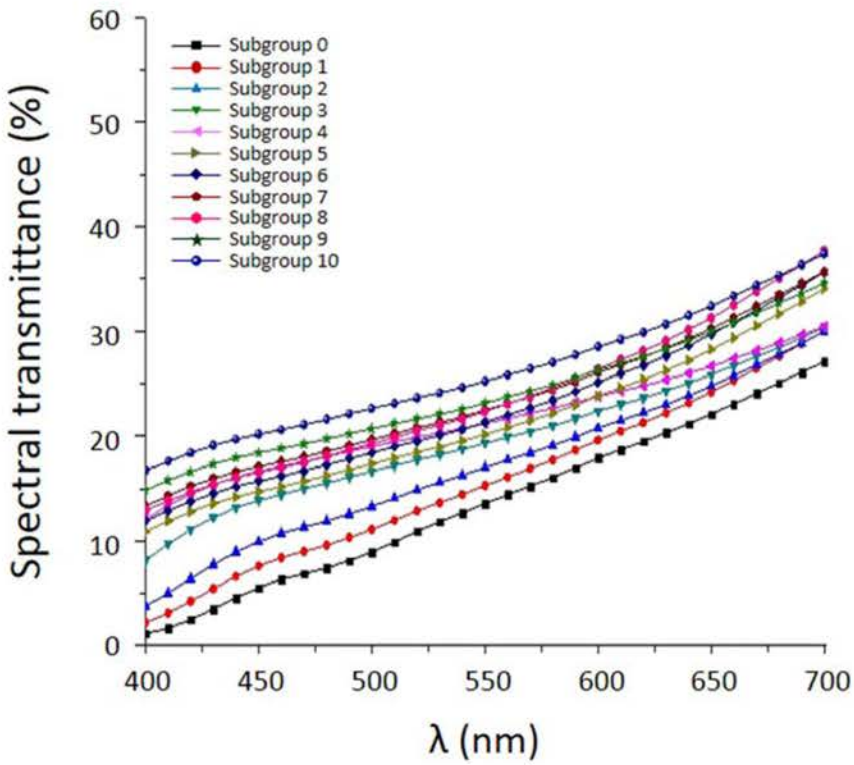

Fig. 58. Spectral reflectance of each subgroup in Group V (Experiment III).

700 nm and transmittance generally increased with increasing the amount of thickness reduction in all groups.

4. DISCUSSION

The effect of various surface modifications on the optical properties of monolithic zirconia materials was evaluated in this study. According to the result of this *in vitro* study, the null hypothesis for Experiment I, II and III could be rejected, because there were significant differences in optical properties between monolithic zirconia with different number of coloring liquid applications, different surface treatments and different amount of thickness reduction.

Based on a study by Lee *et al*. [34], CIE L^*, a^* and b^* values, relative to the standard illuminant D65, of the VITA A2 shade tab, were 52.8 ± 0.2, 0.0 ± 0.0 and 7.8 ± 0.2, respectively. In another study [35], those values of the A2 veneer-layered ceramic cores were 61.2 to 65.8, −0.5 to 1.1 and 8.8 to 12.3, respectively. According to the results of the study by Pecho *et al*. [36], those values of 0.5 mm thick human dentin were 73.3 ± 2.3, −2.1 ± 0.2 and 9.1 ± 1.2, respectively. In the present study, Table 5 shows that CIE L^*, a^* and b^* values of *circa* 2 mm thickness of control group with reflectance mode over the black background were 85.52 ± 0.33, −1.39 ± 0.94 and −1.37 ± 0.08, respectively. Thus, monolithic zirconia material is more whitish and less yellowish appearance compared to veneered ceramic [35] or natural teeth [36].

Based on the result of the present study (Table 5, 9 and 13), at least four times of coloring liquid applications might exhibit similar color values to veneered ceramic [35] or natural teeth [36]. However, direct comparison would be difficult due to different measurement protocol and material thickness.

Cho *et al*. [37] investigated the color and translucency changes of enamel porcelain after repeated staining procedures. The results showed that lightness and chroma increased, but translucency generally decreased after repeated staining. The amount of increase was dependent upon the type of stains and the number of staining cycles. Shah *et al*. [38] investigated the effect of cerium and bismuth coloring salt solutions on the color of 3Y-TZP. A perceptible color difference ($\Delta E^*_{ab} > 1$) was obtained for all test groups, where more yellowish color was identified compared to the control group. The results of the present study likewise exhibited that the increased number of coloring liquid applications with a single shade of A2 reduced lightness and increased yellowish appearance of monolithic zirconia specimen.

Color can be modified by various optical properties, such as reflection, scattering, transmission, refraction and absorption. Furthermore, surface gloss and fluorescence can also have an effect on color modifications [39]. With regard to surface texture, smooth surface could induce more light reflection [39], whereas rough surface could cause the deviation of the

reflection of specular component [40]. Obregon *et al.* [39] investigated the porcelain samples with different degree of surface roughness on the color shift. They demonstrated that different surface textures produced significant differences in hue, chroma and value. Value represented the most significant changes following the modification of surface texture with the smooth surface increasing the value. In addition, there was a shift in hue toward the yellow-red scale with the highly glazed surface. Chung [41] evaluated the effect of polishing procedures on the color and surface roughness of resin composite. In his study, polishing procedures produced a decrease in surface roughness and an increase in lightness value. Lee *et al.* [40] evaluated the effect of surface conditions on the color of dental resin composites with two different measuring geometries, i.e., SCI and SCE. They found that CIE L^* values increased after polishing with the SCE. In the study of Kim *et al.* [23], surface topography influenced especially CIE L^* value of porcelain specimens. CIE L^* value of glazed surface was lower than that of polished surface, whereas CIE a^* and b^* values increased after glazing. Color differences between polished and glazed surface were clinically perceptible ($\Delta E^*_{ab} > 3.7$). In the present study, CIE L^* values decreased after polishing and glazing. CIE L^* values showed the lowest values after polishing even though there were no statistically significant differences between polishing and glazing in some groups. For several studies with resin composites [40, 41] and feldspathic porcelains [23, 39], polishing or glazing procedures resulted in smooth

surfaces which could reflect a greater amount of light than a rough surface. As a result of reflection of incident light, lightness value increased [42]. On the other hand, in the present study, polishing or glazing decreased lightness value. Light scattering could be an important optical characteristic for translucent materials [43] (Fig. 59). Zirconia is polycrystalline structure which can induce maximum scattering effect [18] and thus, zirconia has an opaque appearance to visible light. Based on the results of the present study, surface treatments, such as polishing and glazing, seemed to reduce light scattering on the zirconia surface. Therefore, spectral reflectance decreased after polishing or glazing and lightness value decreased accordingly.

In the present study, polishing or glazing demonstrated a small shift in CIE a^* value toward green which is contrary to the previous reports [23, 39]. There was no statistical difference in CIE a^* value between polishing and glazing in Group I, II and III, while polishing showed lower CIE a^* value than glazing in Group IV and V.

In the present study, glazing increased yellowness when the number of coloring liquid applications was beyond two times. Contrary to glazing, polishing exhibited relatively stable yellow-blue color axis. Glazing procedure demonstrated more color deviation which might be related with any chemical breakdown at elevated temperature [39]. Additional firing

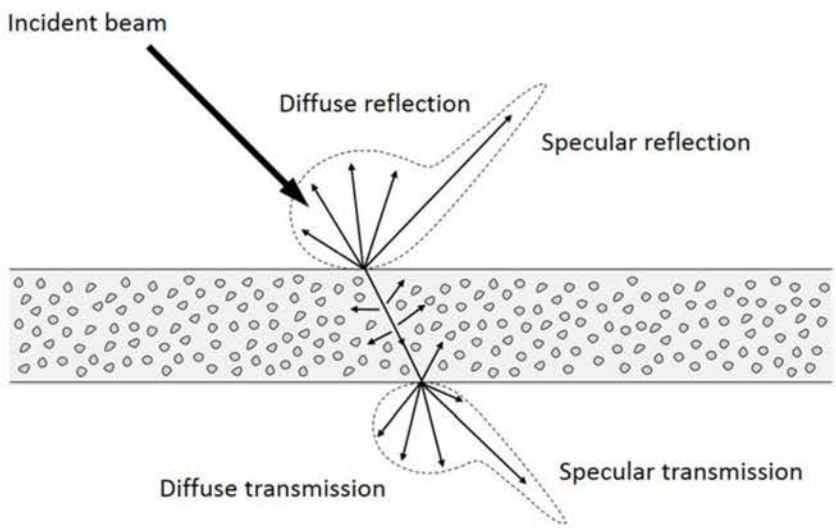

Fig. 59. Overall effect of small-particle scattering for translucent materials.

seems to cause any structural changes of monolithic zirconia. However, this needs to be evaluated in further studies. Moreover, the degree of glossiness after glazing can be controlled either by firing time or by the furnace temperature [44]. Modification of color after glazing might be different depending on the different glazing procedure.

According to the results of the present study, there were highly significant correlations between CIE b^* value and each subgroup as a function of the number of coloring liquid applications. There were negative correlations between CIE L^* value and each subgroup, whereas there were no significant correlations between CIE a^* value and Subgroup N and G. Hence, similar to the result of Experiment I, the lightness decreased and the yellowness increased as the number of coloring liquid applications increased. In addition, this tendency was not changed even after polishing or glazing procedure.

The effect of various changes in thickness of each layer on the final appearance of layered metal-ceramic structure has been studied. Jorgenson and Goodkind [45] evaluated the effect of thickness of porcelain and number of firing on color effectiveness using 1, 2 and 3 mm thickness of porcelain. In their study, no significant difference was found between hue and chroma, whereas value changes were clearly significant with the different thicknesses. As the thickness of the porcelain increased, the value increased. They

reported that the graying effect of opaque layer decreased and effect of the translucency of porcelain increased with the increase of porcelain thickness. Jacobs *et al*. [46] evaluated changes in hue, value and chroma by varying the type of metal-ceramic alloy, the dentin porcelain thickness, and the porcelain shade. They reported that there were visually and spectophtometrically differences between samples of different thickness. However, the direction of changes in value and chroma was dependent on shade and the type of metal-ceramic alloy. Terada *et al*. [25] reported color change with different thickness of dentin porcelain for metal-ceramic. They concluded that L^* value decreased and a^* and b^* value increased as the thickness of dentin porcelain increased. In the study of Lund *et al*. [47] to compare the color of textured opaque porcelains with conventional smooth surface opaque, L^* value increased and b^* value decreased with the increase of the thickness of body and incisal porcelain for both opaque porcelains in metal-ceramic systems. Douglas and Przybylska [2] investigated a dependence on thickness of dentin porcelain for shade matching using different type of dental porcelain systems and shades. They demonstrated that L^* value substantially increased with the decrease of porcelain thickness for metal ceramic systems and In-Ceram alumina system. Increasing the thickness of dentin porcelain produced more scattering and absorption of the incident light and thus, less light reflected back from the opaque layer. In their study [2], the shift in b^* value was considerably sensitive to differences in ceramic thickness, whereas the shift

in a^* value was minimal which is in accordance with the result of the present study.

In the case of metal-ceramic, light scattering and absorption within dentin porcelain layer and specular reflection at the opaque porcelain layer can affect overall color of restorations. Thus, the thickness of dentin porcelain could influence the amount of light at the opaque layer and as a result, the light reflection on the porcelain surface and back at the opaque layer could induce L^* value of the restorations.

There were several studies investigated the effect of the thickness using all-ceramic specimens. Dozic *et al.* [28] determined the effect of different thickness ratio of opaque and translucent porcelain on the overall color of all-ceramic specimens for A1, A2 and A3 shade. Both a^* and b^* values increased as the thickness of opaque layer increased for all shades. L^* value was shade dependent. Shokry *et al.* [27] investigated the effect of the different thickness ratio of core and veneer of two all-ceramic systems, such as IPS Empress and In-Ceram Spinell, on the color parameters of layered specimens. In their study, L^* value decreased while a^* and b^* values increased as the total specimen thickness increased for both ceramic systems. In addition, core and veneer interaction as well as the thickness strongly influenced overall color of layered ceramic specimens. They explained that increased absorption of

incident light with thicker specimens reflected reduced quantity of light and accordingly, L^* value decreased. Furthermore, they demonstrated that translucency of dental ceramic was often related with lower L^* value. Ozturk *et al*. [29] evaluated the effects of various dentin ceramic thicknesses (0.5, 1 or 1.5 mm) with a core thickness of 1 mm and number of firings on the color of lithium disilicate glass-ceramic and zirconium oxide all-ceramic systems. In their study, L^* value significantly decreased as the veneering ceramic thickness increased for IPS e.max Press and DC-Zirkon specimens. There were significant increases in a^* and b^* values with the increase of the thickness for IPS e.max Press specimens. For DC-Zirkon specimens, there was a significant increase in a^* value, while no significant difference in b^* value as the ceramic thickness increased. The present study has shown that there was a significant decrease in L^* value after initial 0.1 mm reduction, but there were no distinct changes from 0.2 mm to 1.0 mm reduction in most groups.

With regard to all-ceramic restorations, the amount of light reflection at the opaque core seems to be different between all-ceramic systems with different core translucency although L^* value generally decreased with the increased thickness of veneering porcelain due to the increased absorption of incident light. Douglas and Przybylska [2] demonstrated that due to the lack of opaque

core, the semi-translucent all-ceramic porcelain systems were less affected by reduced porcelain thickness compared to metal-ceramic for shade matching.

Incident light that is not reflected at the surface layer suffer internal reflection by reversing the direction and may be seen by the inspector [48] (Fig. 60). In the present study, the changes in L^* value showed different aspect compared to metal-ceramic and all-ceramic systems. The possible reason would be the difference of layering structure, since monolithic zirconia consists of a single opaque layer. Based on the results of the present study, it can be inferred that there might be reduced scattering due to the reduced thickness which induced lower L^* value at first 0.1 mm reduction. However, as the thickness reduction proceed, monolithic zirconia itself acts as an opaque core and could induce internal reflection. With the increase of the thickness reduction, reduced reflection might compensate for increased internal reflection and thus, L^* value could be relatively stable up to 1.0 mm reduction ($-0.59 < r < 0.35$, $R^2 < 0.35$, Fig. 40). Colorant might affect scattering and absorption *circa* 0.4 mm deep in the specimens and there seems to be no additional colorant effect on L^* value (Fig. 43). Consequently, clinicians can predict that there might be a small decrease in L^* value after initial reduction in a clinical situation, but L^* value could remain stable until 1.0 mm of thickness reduction. In addition, further study should be required to determine whether there is any difference in L^* value when the thickness reduction is more than 1.0 mm.

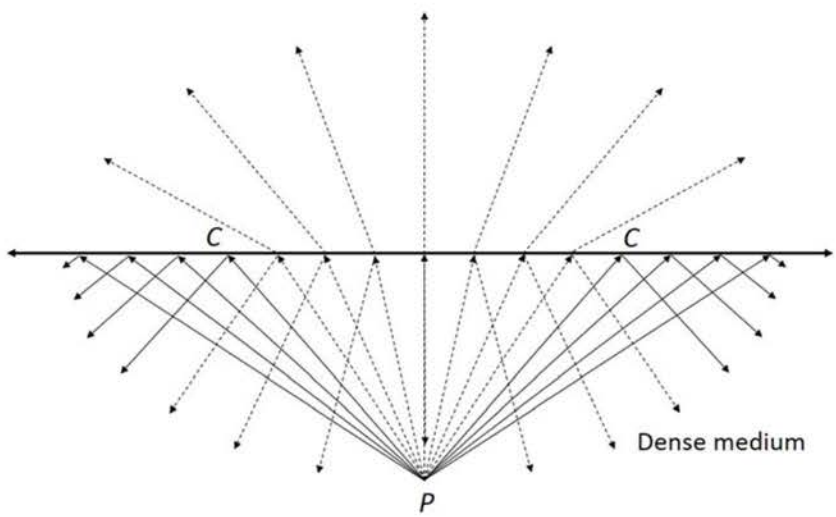

Fig. 60. When a light passes from a material with a higher refractive index to a lower refractive index, total internal reflection can occur. *P*: pigment particle. *C*: critical angle.

Chromatically, a^* value substantially increased, while b^* value generally decreased with the decrease of the thickness. In terms of the shift in chroma, b^* value was more sensitive to the change of thickness than a^* value showing minimal shifts in a^* value. This result is in accordance with previous studies [2, 28, 29]. According to the study of Douglas and Brewer [49], dental observers were more sensitive and critical to the color difference in redness than yellowness for metal-ceramic crowns. Therefore, perception of color difference in yellowness might be somewhat tolerant toward thickness changes compared to redness for human observer. However, the previous study [49] was conducted with metal-ceramic crowns and therefore, further study should be performed to determine whether there is any difference in subjective color assessment between a^* and b^* value for monolithic zirconia restorations.

As a result, it can be inferred that the color of ceramic restoration is influenced by its thickness regardless of the ceramic systems and the direction of the changes of L^*, a^* or b^* value was dependent on the type of the material tested. In the present study (Fig. 44 and 45), Group I joined with other graphs at 0.1 mm reduction, and Group II joined at 0.2 mm reduction, and Group III joined at 0.3 mm reduction, and Group IV joined at 0.6 mm reduction, and Group V joined at 0.6 mm reduction for both a^* and b^* value. Based on the results of the present study, it can be inferred that one time of application of coloring

liquid could infiltrate 0.1 mm deep through monolithic zirconia, two times of application could infiltrate 0.2 mm deep, three times of application could infiltrate 0.3 mm deep, four times of application could infiltrate 0.6 mm deep, five times of application could infiltrate 0.6 mm deep through monolithic zirconia.

Several studies [50-54] have attempted to investigate the threshold for perceptibility and acceptability of color difference. Johnston and Kao [50] determined 3.7 ΔE^* units as a perceptibility threshold and 6.8 ΔE^* units as a borderline for color match or mismatch between composite veneers and teeth. Other *in vivo* study [51] indicated 2.6 ΔE^* units as 50/50 perceptibility of color difference. This perceptibility threshold was different from those of other *in vitro* studies which identified 1 ΔE^* unit [52] and 2 ΔE^* units [53]. Ghinea *et al*. [54] reported that a 50% perceptibility threshold was 1.8 ΔE^* units using dental ceramic discs. The interpretation of the color difference for the present study is based on the visual matching study of Johnston and Kao [50].

In the present study, color differences for all colored groups when compared to the control group in Experiment I were above a clinically perceptible level ($\Delta E^*_{ab} > 3.7$), while there were no perceptible color changes between two subsequent groups.

Color differences between no treatment and polishing was higher than between no treatment and glazing in Experiment II of the present study. This would be caused by the higher difference of lightness value between no treatment and polishing. This is in accordance with Chung's study [41] which demonstrated that color difference was mainly determined by the lightness rather than the hue and chroma. Color differences between no treatment and polishing can be perceived in a clinical setting ($\Delta E^*_{ab} > 3.7$). Color difference between no treatment and glazing can also be detectable in a clinical setting. Thus, surface treatment, whether polishing or glazing, could modify the color interpretation. However, there were no perceptible color differences between polishing and glazing in most groups, which means that human eye cannot detect the color difference between these two procedures.

Based on the results of Experiment III in the present study, color differences can be perceived in a clinical setting ($\Delta E^*_{ab} > 3.7$) even after first 0.1 mm reduction regardless of the number of coloring liquid applications. In addition, when the thickness was reduced by 0.3 mm or more, larger color difference compared with no reduction was perceived as the number of coloring liquid applications increased (Fig. 51).

Consequently, various surface modifications, such as coloring procedure, polishing or glazing, and thickness reduction could induce noticeable shift in

color parameters. Since high value of monolithic zirconia yield a less vital appearance, reduced value resulted from coloring, polishing and glazing might improve natural-looking appearance. Glazing could improve yellowish appearance of monolithic zirconia. However, loss of glaze layer could occur within the first six months after insertion of the restorations [55]. Since there was no perceptible color difference between polishing and glazing, polishing could be recommended which seems to be more stable in terms of long-term color appearance. Thickness reduction could reduce brightness and produce reddish and bluish appearance of monolithic zirconia. Therefore, this tendency should be taken into account during the adjustment procedure by the dentist to achieve an optimal occlusal contact. In addition, thickness reduction could induce wider range of the shift in b^* values than in a^* values and thus, this should be considered in color matching as well.

In the present study, the statistical analyses showed that the influence of the number of coloring liquid applications, polishing and glazing on TP value was not significant. There was a study [56] which showed that there were significant differences in contrast ratios of Procera zirconia between specific shades. Pecho *et al*. [36] evaluated the translucency of both non-colored and colored zirconia, and compared them with human and bovine dentin of 0.5 mm thickness. They exhibited that TP values of human dentin, bovine dentin and zirconia showed no significant differences among them, indicating

17.2 ± 1.8 for human dentin and 17.0 ± 1.7 for bovine dentin. No significant differences were also found between non-colored and colored zirconia systems. Yu *et al*. [57] investigated the translucency of human and bovine enamel and dentin. Mean TP values of 1 mm thick bovine enamel, bovine dentin, human enamel and human dentin were 14.7, 15.2, 18.7 and 16.4, respectively. In the present study, TP values of monolithic zirconia specimens of 2 mm in thickness ranged from 9.15 to 11.69 in Experiment I. Experiment II and III exhibited lower TP values than Experiment I and this might be due to the edge loss effect of the spectrophotometer. The configuration of spectrophotometer is important in the measurement of color and translucency. Bolt *et al*. [58] and ten Bosch and Coops [59] described that the re-emitted photon was scattered beyond the edge of the opening especially for translucent material, and the smaller window area view caused the greater amount of edge-loss. In an attempt to reduce the edge-loss, the window size of 19 mm in diameter and the reduced beam size of 1 mm × 5 mm were used in Experiment I of the present study.

Jiang *et al.* [60] reported that sintering temperature and particle size significantly affected the density and transmittance of Y-TZP and the smaller nanoparticles had higher transmittance than that of larger particles. In addition, the presence of impurities and the sintering conditions, such as sintering temperature and time, could affect significantly on the grain size [61, 62].

There have been several sintering methods which have tried to derive nanocrystalline ceramic with a full density, such as spark-plasma-sintering [63], hot pressing [64], and two-step sintering procedure [65], etc. Casolco *et al*. [66] had attempted to obtain translucent zirconia ceramics combining nanopowders with nanocrystalline grain sizes of full density. They suggested that when the grain size is significantly smaller than the wavelength of visible light, there would be more transmission of light rather than a scattering caused by interaction with internal particles.

Based on the results of the present study, translucency of monolithic zirconia is dependent on the thickness, and light transmission is a function of its thickness as well. The present study measured total transmission including scattering using the spectrophotometer with an integrating sphere [67]. Fig. 32-39 and Fig. 54-58 showed that transmittance generally increased with the increase of incident wavelength. As stated by the Rayleigh scattering equation [68], scattering of incident light seems to decrease in monolithic zirconia with increasing wavelength.

The effect of the thickness on the translucency of ceramic restorations was studied in all-ceramic systems. Antonson and Anusavice [30] investigated the contrast ratio of dental core and veneering ceramics as a function of ceramic thickness. There was a relatively strong positive linear correlation between

CR and thickness ($R^2 > 0.81$). Hefffernan *et al*. investigated the effect of the thickness of a core material [43] on its translucency, and the thickness of a veneered core material [69] on the overall translucency of the specimens. They demonstrated that increased thickness resulted in greater opacity. O'Keefe *et al.* [70] suggested that that the thickness of the porcelain veneer was the primary factor affecting light transmission and not the opacity. The present study demonstrated similar results. TP values generally increased as the amount of thickness reduction increased in all groups ($r > 0.94$, $R^2 > 0.89$, $P < 0.001$, Fig. 53). Based on Lambert's law which states that equal amounts of absorption occur in equal thicknesses of material [68], decreasing the thickness of material allow greater amount of light transmission. Furthermore, the fraction of incident light that are reflected, absorbed, and transmitted depend on the thickness of the specimen as well as the scattering and absorption characteristics [71]. Accordingly, translucency of monolithic zirconia restoration could be controlled by its thickness as well as the application of efficient sintering procedures and grain size [60, 63-66] rather than coloring procedure, polishing or glazing.

Opalescence can improve the natural appearance and the vitality of restorations. For opalescence feature, there should be a light scattering of shorter wavelengths of the visible spectrum in a translucent material. Opalescent material appears blue in reflected light and orange-yellow in

transmitted light [72]. Based on the previous study [73], OP value of the commercial resin composite specimens of 1 mm in thickness was in the range from 5.7 to 23.7, which was varied by their brand and shades. Another study [74] reported that OP values of bovine enamel of 0.7 mm-1.1 mm in thickness ranged from 7.6 to 22.7, which was varied by the configuration of spectrophotometers and those of human enamel of 0.9 mm-1.3 mm in thickness ranged from 19.8 to 27.6. Cho *et al.* [75] investigated the opalescence of all-ceramic core and veneer materials using a spectrophotometer. In their study, the range of OP value was 1.6-6.1, 2.0-7.1, 1.3-5.0 and 1.6-4.2 for the core, veneer, A2- and A3-layered specimens, respectively, and all of which were significantly influenced by the type of materials. According to one of the US patents [33], the OP value was represented based on 1 mm thick resin composite specimens as a difference in the chromaticity [$\Delta C^*_{ab} = (\Delta a^{*2} + \Delta b^{*2})^{1/2}$] between the reflected and transmitted colors. It was reported that the OP value should be at least *circa* 9 to contribute the vitality of dental restorative composites. The restorative materials with OP values of between 4 and 9 may be considered to have some opalescence only slightly discernible to the naked eyes. Based on the results of the present study, when the number of coloring liquid applications is more than three times, monolithic zirconia restorations could be regarded non-opalescent under this criterion.

According to the results of the present study, there was a highly significant correlation between OP and Δb^* ($r = -0.782$). This means that OP was mainly influenced by the change in CIE b^*. This is in accordance with the several studies [74, 76] on opalescence, which demonstrated that OP value was correlated with Δb^* and ΔE^*_{ab}. However, there is a study [75] which showed that the correlations between OP and ΔE^*_{ab}, or between OP and Δb^* were not significant for layered all ceramic specimens. On the other hand, another study [73] exhibited that there was a weak correlation between OP and Δa^* ($R^2 = 0.076$). Several studies [73, 77] demonstrated that there was a strong correlation between TP and OP. However, no correlation was found in the present study. Since the presence of small internal particles influences maximizing opalescence in a translucent material [78], grain size might have a significant effect on the translucency and opalescence of monolithic zirconia restorations.

Esthetic demand is increasing for monolithic zirconia restorations. Fabricating monolithic zirconia restorations with an appropriate shade, translucency and opalescence is becoming a major concern for a natural-looking appearance. Surface modifications to improve esthetic outcome, such as coloring procedure, polishing and glazing, and thickness reduction followed by adjustment procedure could be possible factors that affect overall appearance of monolithic zirconia restorations. Considering the prediction

and reproduction of overall appearance of monolithic zirconia restorations, clinicians should take into account the possible color deviations after coloring procedure, polishing or glazing, and thickness reduction. Furthermore, different shade guides considering any color changes following surface modifications can be helpful for monolithic zirconia restorations.

This *in vitro* study has several potential limitations. The first limitation is that even application of coloring liquid on the specimens, uniform application of glazing paste without any void, and exact amount of the increase of coloring liquid applications were difficult to control. Secondly, possible pressure fluctuation inside the furnace during sintering process might induce uneven color of the specimens. In addition, possible temperature fluctuation inside the furnace might not yield homogenous glazing. Thirdly, the aperture diameter of spectrophotometer used for Experiment II and III in the present study for reflectance measurement was 3 mm and possible edge loss would affect color measurement. Finally, this study was conducted with limited color of shade A2 and with a specific kind of monolithic zirconia system and coloring liquid. Therefore, the influence of varied color combinations with different monolithic zirconia systems should be further studied. In addition, further study is needed to investigate the effect of additional surface treatment after thickness reduction by the adjustment procedure on the color and translucency of monolithic zirconia restorations. Moreover, further study

should consider *in-vivo* overall color shift which might be related with the color of underlying cement and tooth substrate.

For the clinical application of monolithic zirconia, based on the results of the present study, at least four times application of coloring liquid could be recommended considering the change of the optical properties according to the surface modifications. Especially, for the application in anterior region, additional blue coloring could be considered since monolithic zirconia has little opalescence property. In addition, polishing would be recommended rather than glazing for monolithic zirconia restorations concerning long-term color stability. Furthermore, the thickness of monolithic zirconia restorations could be determined considering the translucency especially for the discoloration of the tooth substructure.

5. CONCLUSION

Within the limitations of this study, the following conclusions can be drawn. Increasing the number of coloring liquid applications results in a decrease of the lightness and an increase of the yellowish appearance of monolithic zirconia. Polishing decreases the lightness and glazing also decreases the lightness, while it increases the yellowish appearance of monolithic zirconia. Increasing thickness reduction decreases lightness and increases the reddish appearance and decrease the yellowish appearance of monolithic zirconia. In addition, reduced thickness produces more translucent monolithic zirconia. Clinicians can predict that there would be a shift in optical properties of monolithic zirconia restorations with various surface modifications, such as coloring, polishing, glazing and thickness reduction.

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국문 초록

다양한 표면 변화가 치과용 단일 구조 지르코니아 수복물의 광학적 특성에 미치는 효과에 관한 연구

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연구 목적: 다양한 표면 변화가 단일 구조 지르코니아의 광학적 특성에 미치는 효과에 관하여 알아보고자 하였다; 1) 염색 횟수가 단일 구조 지르코니아의 광학적 특성에 미치는 효과, 2) 연마와 광택 처리가 단일 구조 지르코니아의 색상과 투명도에 미치는 효과, 3) 다양한 삭제량이 단일 구조 지르코니아의 색상과 투명도에 미치는 효과.

재료 및 방법: 단일 구조 지르코니아로 BruxZir, 염색액으로 A2 색상의 Tanaka ZirColor를 선택하였다. 첫 번째 실험에서는 총 18개의 지르코니아 시편(가로 27.6mm, 세로 27.6mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 6개의 그룹(그룹 O 에서 그룹 V)으로 나누고 각각 색상, 색 차이, 투명도 및 유백광도를 측정하였다. 두 번째 실험에서는 총 45개의 지르코니아 시편(가로 16.3mm, 세로 16.4 mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 5개의 그룹(그룹 I 에서 그룹 V)으로 나누고, 각 그룹을 표면 처리 방법에 따라 3개의 소그룹(대조군(N), 연마군(P), 광택군(G))으로 나눈 후, 각 시편의 색상, 색 차이 및 투명도를 측정하였다. 세 번째 실험에서는 총 165개의 지르코니아 시편(가로 16.3mm, 세로 16.3mm, 두께 2.0mm)을 염색액을 이용한 염색 횟수에 따라 5개의 그룹(그룹 I 에서 그룹 V)으로 나누고, 각 그룹을 두께 삭제량에 따라 0.1mm 간격으로 1.0mm 삭제까지 11개의 소그룹(소그룹 0 에서 소그룹 10)으로 나눈 후, 각 시편의 색상, 색 차이 및 투명도를 측

정하였다. 모든 측정은 CIELAB 색공간을 사용하였고, CIE 표준 광원 D65에서 분광광도계를 사용하여 한 시편 당 다섯 부위를 측정하였다. 통계는 SPSS 프로그램을 이용하여 일원분산분석 및 Scheffé 다중검정을 실시하였으며, 각각의 측정값 간의 유의성 및 상관관계도 알아보았다 (a = 0.05).

결 과: 첫 번째 실험에서는 염색 횟수가 증가함에 따라 CIE L^* 값과 유백광도는 감소하였으며, CIE b^* 값은 증가하였다. CIELAB 색차는 1.3 에서 15.7 사이로 나타났으며, 투명도의 유의한 변화는 없었다. 두 번째 실험에서는 모든 그룹에서 N과 P간에 CIE L^* 값의 유의한 차이를 보였으며, P와 G간에 CIE b^* 값의 유의한 차이를 보였다. 모든 그룹에서 N과 P간의 색 차이를 지각할 수 있었으며, 대부분의 그룹에서 N과 G간의 색 차이를 지각할 수 있었다($\Delta E^*_{ab} > 3.7$). 대부분의 그룹에서 각 소그룹 간의 투명도의 차이는 나타나지 않았다. 세 번째 실험에서는 삭제량의 증가에 따라 CIE L^* 과 CIE b^* 값이 감소하였으며, CIE a^* 값은 증가하였다. 모든 그룹에서 소그룹 0과 1간의 색 차이를 지각할 수 있었으며, 모든 그룹에서 삭제량의 증가에 따라 투명도가 증가하였다.

결 론: 염색 횟수의 증가는 단일 구조 지르코니아의 명도를 감소시키고, 황색 빛을 증가시킨다. 연마 처리는 명도를 감소시키고, 광택 처리는 명도를 감소시킬 뿐만 아니라 황색 빛을 증가시킨다. 삭제량의 증가는 명도를 감소시키고, 붉은 빛 및 푸른 빛을 증가시킨다. 삭제량의 증가는 투명도를 증가시킨다.

· **주요어:** 단일 구조 지르코니아, 염색액, 색상, 투명도, 표면 특성 변화
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